Durability and physical characterization of anti-fogging solution for 3D-printed clear masks and face shields

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Background. The COVID-19 pandemic brought forth the crucial roles of personal protective equipment (PPE) such as face masks and shields. Additive manufacturing with 3D printing enabled customization and generation of transparent PPEs. However, these devices were prone to condensation from normal breathing. This study was motivated to seek a safe, non-toxic, and durable anti-fogging solution. **Methods**. We used additive 3D printing to generate the testing apparatus for contact angle, sliding angle, and surface contact testing. We examined several formulations of carnauba wax to beeswax in different solvents and spray-coated them on PETG transparent sheets for testing contact and sliding angle, and optical clarity. Further, the integrity of this surface following several disinfection methods such as soap, Isopropyl Alcohol, or water alone with gauze, paper towels, and microfiber, along with disinfectant wipes, was assessed. **Results**. The results indicate a 1 : 2 ratio of carnauba to beeswax in Acetone optimally generated a superhydrophobic surface (contact angle 150.3 + 2.1 ° and sliding angle 13.7 + 2.1 °) with maximal optical clarity. The use of soap for disinfection resulted in the complete removal of the anti-fogging coating, while Isopropyl Alcohol and gauze optimally maintained the integrity of the coated surface. Finally, the contact surface testing apparatus generated a light touch (5000 N/m^2) that demonstrated good integrity of the antifogging surface. These results suggest that the routine use of clear PPEs could be further enabled by using a novel anti-fogging solution.

Durability and Physical Characterization of Anti-Fogging Solution for 3D-Printed Clear Masks and Face Shields

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ABSTRACT

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Methods. We used additive 3D printing to generate the testing apparatus for contact angle, sliding angle, and surface contact testing. We examined several formulations of carnauba wax to beeswax in different solvents and spray-coated them on PETG transparent sheets for testing contact and sliding angle, and optical clarity. Further, the integrity of this surface following several disinfection methods such as soap, Isopropyl Alcohol, or water alone with gauze, paper towels, and microfiber, along with disinfectant wipes, was assessed.

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INTRODUCTION

The COVID-19 pandemic has had a significant toll on the public health, biomedical, and financial aspects that have significantly changed society.(1,2) Among various healthcare services, the presence of SARS-CoV in the nasal and upper respiratory tract presented significant challenges for dentists and anesthetists. Clinical dentistry presented a significant challenge due to the proximity and inherent aerosol-generating procedures. (3-5) The use of personal protection equipment (PPE) such as face masks and face shields was integral in reducing the spread of infections among the general population. (6) While dentistry routinely utilizes face masks, double masking, and the impervious nature of N95 further compounded the pandemic fear and anxiety in patients naturally apprehensive about their dental visits. (7-9) Another major limitation of these masks is that they hindered communication due to the lack of non-verbal cues such as facial expressions, lip and tongue movements. (10-12) This is particularly challenging in differentially ables (hearing impaired) patients due to voice attenuation with increased ambient noise in dental operatories. (13-16) The use of transparent masks and face shields has been demonstrated to improve these limitations. (17)

Another major challenge during the early phase of the pandemic was the significant disruption of the PPE supply chain requiring local innovations where additive manufacturing using 3D printing came to the forefront. (18-21) The ability to generate required quantities based on need and additional customization for individual applications were significant advantages of this approach. (22) Building on this, in collaboration with e-NABLE, a 3D printing community, students at the University at Buffalo School of Dental Medicine generated transparent PPEs using 3D printing and vacuum casting. Among them, several custom designs were specifically generated to accommodate commercially available dental loupes that are made available freely online.(23) These clear masks and face shields were well accepted and generated much enthusiasm among clinical users. Besides dentistry, another group of clinicians that notably adopted these clear PPEs were the local speech and hearing-impaired clinics. However, an immediate limitation of this approach reported by users was the fogging due to the moisture from regular breathing. This essentially negated the advantage of the transparent nature of these masks and face shields and presented additional hindrance of needing repetitive wipe-downs.

The commercially available anti-fogging solutions used for eyeglasses and car exteriors contain polydimethylsiloxane and silica nanoparticles. The long-term use of these solutions was considered unsuitable due to their potential for skin irritation, and potential inhalation or ingestion. Hence, we sought to identify a non-toxic, antifogging coating for the transparent masks and face shields for long-term use. To design such a solution, specific design criteria needed to be met. The surface of a water droplet is attracted to the bulk of the droplet due to cohesion which results in their beading. The shape that a drop takes on a given surface depends on the surface tension of the fluid and the physical characteristics of the surface. A surface-fluid interface is the contact angle that measures the wettability of a surface. Contact angles can vary from hydrophilic (less than or equal to 30°), hydrophobic (120°), or superhydrophobic (over 150°) where the higher the contact angle, the lower its wettability. (24) Another measure of the extent of moisture retention on a surface is the sliding angle which is measured by tilting the surface with the solution bead to determine the angle at which it rolls off. The more hydrophobic the surface, the higher the angle of loss of fluid droplets.(25) Another key design aspect of the anti-fogging solution would be its resistance to wear due to repeated skin surface contact during mask use. The ideal solution would offer a thin, durable coating for daily use that would not need repetitive applications while maintaining maximal transparency.

Natural products offer attractive non-toxic coatings. Prior studies have examined the combination of cinnamon and nutmeg that produces high hydrophobicity. (26) Although both are natural products, there are some toxicity concerns due to high cuprous oxide. Drawing inspiration from the naturally-occurring, extremely hydrophobic lotus leaves that expel water due to their micro-structural features termed papillae that exude epicuticular waxes, cutin. (27,28) Alternatively, Carnauba and beeswax have also been shown to achieve high hydrophobicity. (29) With high hydrophobicity, the solution was intended for food containers to prevent waste. However, there was a lack of clarity in the concentrations used and the resulting hydrophobicity. This study examined the different formulations of these two waxes together and evaluated the contact angle, sliding angle, and optical clarity. The optimized formulation was subjected to durability testing with manual disinfectant procedures and long-term contact-wear testing.

MATERIALS AND METHODS

Formulation of anti-fogging solutions

Hydrophobic wax coatings were generated in acetone or methanol as solvents using ultrasonication. Different ratios of carnauba wax to beeswax were employed namely 0.4375 g: 0.4375 g (1:1), 0.35 g: 0.525 g (1:1.5), 0.33 g: 0.66 g (1:2), 0.417 g: 0.834 g (1:2) were melted in a 50 ml plastic tube (Corning, Thermofisher Waltham, MA) in a water bath. After the waxes had melted, 25 milliliters of either acetone or methanol (both Sigma Aldrich, St. Louis, MO) were added, and the solution was emulsified immediately using ultrasonication (Q2000, QSonica, Newtown CT) at 90% amplitude for 3 minutes and transferred into a 2 oz glass spray bottle.

Contact and Sliding Angle testing

Solutions were sprayed onto a 2 x 2-inch sheet of PETG at roughly a distance of 5 inches, spraying 10 times. After drying, the sheets of PETG were tested for contact angle, sliding angle, and transmission. A custom contact and sliding angle device were generated based on commercially available models using online CAD software (Onshape, PTC, Rockwell Automation, Boston MA). The devices were printed using Polylactic acid (PLA) filament (Overture, Overture 3D Technologies, Texas) on a i3 Prusa 3D printer (Prussa Research, Prague Czech Republic) (**Figs. 1 A - C**). The device included a slot for to hold a mobile phone to take digital pictures of the droplet. After fixing the plastic surface to the platform, droplets of water were generated with a 3 ml syringe and 14-gauge needle and digital image were captured for analysis (**Fig. 1 D**). The digital images were analyzed using the NIH ImageJ (ver. 1.53n) software. The sliding angle set up included a protractor to assess the angle at which the droplet slides off. The platform was tilted slowly until the water droplet slid off, and the angle was documented (**Fig. 1 E**). All studies were performed in replicated and repeated at least twice.

Optical clarity analysis

The laser apparatus consisted of a 650 nm diode laser (Weber Medical, Beverungen, Germany) at 10 mW/cm^2, and a sensor with power meter (both Thor Labs, Newton NJ) (**Fig. 1F**). The sheets of PETG were placed on top of the power sensor, and transmission was assessed.

Routine manual disinfection testing

The PETG coated surfaces were subjected to different solutions such as soap solution (10% v/v, Dawn, Procter & Gamble, Cincinnati OH), Isopropyl alcohol (70%, Sigma Aldrich, St. Louis MO), and water alone using paper towels (Uline, Milton ON), gauge (Henry Schein, New York, NY) or microfibers (Magic Fiber, Miami FL). These disinfectants were applied ten times in a uniform, lateral motion with constant manual pressure by a single operator. Disinfection wipes (Metrex, Orange CA) were used in another group.

Coating Durability following contact wear testing

To simulate accelerated contact wear testing, a custom apparatus was designed to mimic skin contact. Using the CAD software (Onshape, PTC, Rockwell Automation, Boston MA), a rotating platform with spring-loaded bases was designed to apply suitable skin contact forces. (30-32) The device was 3D printed with PLA filament (Overture, Overture 3D Technologies, Texas) on a i3 Prusa 3D printer (Prussa Research, Prague Czech Republic). The wheel was connected to a DC Motor (Greartisan, Shenzen Hotec, China) via a speed controller and a 9V battery which allowed the motor to function up to 1000 RPMs. After the coated samples were mounted on the platforms, the apparatus was positioned to allow the platforms to extend and retract using centrifugal force while spinning, hitting a skin-equivalent surface to simulate contact. To simulate multiple skin contacts, the device was operated for 5 minutes, allowing each platform to contact the wall about 5000 times for each sample. The contact force applied by the rotational, spring-loaded platform was measured with a digital force probe (Baoshishan, China). Following the contact wear testing, samples were assessed for changes in contact angle, sliding angle, optical clarity and examined with ultrastructure analysis.

Ultrastructure analysis

Scanning electron microscopy (SEM) analysis was performed to determine the coating on the plastic sheet samples. Samples were sputter-coated for 120 seconds and examined using SEM (Hitachi, S-4700, Japan) with a voltage of 15 kV.

Statistical Analysis

Data was organized in Excel (Microsoft, Seattle WA) and presented as Means with standard deviations. Data was subjected to a student T test was performed or two-way analysis of variance (ANOVA) among different treatments with Bonferroni's correction for multiple comparisons where appropriate. A $p < 0.05$ was considered statistically significant.

RESULTS

Formulation and Characterization of Anti-Fogging solutions

We first examined different ratios of carnauba and beeswax in two solvents namely Acetone and Ethanol to generate a homogenous solution (**Fig. 2A**). The contact angles of the acetone-based solutions were higher than their methanol counterparts (**Figs. 2B** and **2C**). The coating with the highest contact angle was the 1: 2 ratio in acetone solution with a mean contact angle of $150.3 \pm 2.1\ ^{o}$. This categorizes this surface coating as superhydrophobic and was significantly ($p < 0.05$) more hydrophobic than the uncoated plastic surface. The higher concentrations at 1 : 2 ratio did not significantly improve these characteristics further. As expected the sliding angle for this coating was the lowest at 13.7 ± 2.1^{o} which was significantly ($p < 0.05$) lower than the uncoated plastic surface (**Figs. 2D** and **2E**). The optical clarity of these formulation appears to vary with the concentration of the beeswax component as lower amounts resulted in higher clarity (**Figs. 2F** and **2G**). The methanol-based formulations had a higher degree of optical clarity than the acetone counterparts. Based on these results, we decided to pursue the 1:2 carnauba to bee wax in acetone formulation for subsequent studies.

Effect of routine disinfection procedures on durability of antifogging solution

The use of disinfectants is a necessary part of routine plastic mask and face shield due to the prominent aerosol generating dental procedures. (33-35) Next, we examined routine disinfectant procedures employed in the dental office namely, soapy water, 70% Isopropyl Alcohol with paper towels, gauze, or microfiber, and disinfectant wipes. We noted a significant ($p < 0.05$) reduction in the contact angle after all disinfection methods (**Fig. 3A**). Among all these methods, Isopropyl Alcohol with gauze reduced the contact angle the least ($144.3 \pm 1.6\ ^{o}$) while soap made the surface most hydrophilic (wetable). Concurrently, the sliding angle analysis showed a similar trend, increasing overall with all, but the soap group, disinfection methods (**Fig. 3B**). The Isopropyl alcohol group showed the least change and no statistically significant difference was noted with the paper towel method. Interestingly, the optical clarity appeared to vary after individual disinfectant methods with some showing increases while other showing significant decreases (**Fig. 3C**). The method of disinfection that showed most increase in light transmission was wipes alone while the most reduction was observed with Isopropyl Alcohol and paper towel method. These results suggest that using Isopropryl Alcohol with gauze is optimal for disinfection as it minimally affects antifogging coating properties after repeated use.

Antifogging coating stability after contact wear testing

Finally, we investigated the durability of the anti-fogging solutions as they would be subjected to rigorous daily wear and tear during PPE use. We first created a simple rig to simulate skin contact based on a 3D printed apparatus (**Fig. 4A** and **B**). The rig was designed as a rotating platform that would result in a repetitive contact of the coated device with a stationary leather surface. The force exerted by facemask on the nose bridge and chin contact has been assessed to be 45 to 91 mm of Hg (6000-12,000 N/m^2) (**Fig. 4C**). (30-32) The constant skin contact force in our device was assessed to be 5000 N/m^2 that could be approximated to a repeated light skin contact.

Following the simulated wear, the contact and sliding angle as well as optical clarity was assessed. We observed a significant ($p < 0.05$) reduction in contact angle (133.3 ± 1.5^o) and concomitant increase in the sliding angle (23.7 ± 1.5^o) compared to the uncoated control surfaces. While optical clarity was not significantly different before and after wear simulation, there was a trend towards reduced transmission. Ultrastructure analysis noted the wear simulation resulted in significant accretions and scratches that likely contribute to the reduced light transmission observed. These results indicate the coating will likely need to be replaced over repeated PPE use as often as every other week.

DISCUSSION

The COVID pandemic presented new challenges to healthcare with an urgent need for innovations in disinfection approaches. The use of PPEs was central in the attempts to mitigate the spread of infection and a major part of the solution to mitigate the health crisis. The use of these barriers continues to have an impact on both the psychological, and medical well-being of both the patients and the healthcare professionals themselves.(36) The initial response to the pandemic brought manufacturing and supply chain disruptions resulting in additive 3D printing enthusiasts to offer custom, local solutions. Our team at the University at Buffalo focused on the transparent PPE designs and dental loop attachments as our innovative solution. The condensation from breathing obscured the functions of the clear PPEs that presented a major impasse to their use. This work was specifically motivated to address this deterrent for routine clear PPE use.

We tested two transparent polymers for this analysis namely Polyvinyl chloride (PVC) and polyethylene terephthalate glycol (PETG). PETG is a thermoplastic that has a reputation for its high impact resistance and ductility, offers better chemical resistance and durability, and is commonly used. To generate an anti-fogging solution for our clear face shields and masks, a major design challenge was the close proximity to nose and mouth.(37,38) We chose to pursue natural waxes as the main ingredient as they provided a natural, non-toxic solution that has a well-established track record of biological safety. (39) The other major component constituent was an polar solvent to enhance miscibility and dispersion of these waxes. We chose to examine Acetone and Ethanol in our formulation. The Acetone formulation performed more superiorly to generate the most superhydrophobic product for spray coating. Our initial efforts at dissolving the waxes in Acetone (boiling point of 132.8°F) by heating alone resulted in evaporation and non-uniform dissolution. Hence, we chose to utilize ultrasonication that resulted in an effective homogenous solution. Among the various formulations, the most superhydrophobic surface was generated by the 1 : 2 ratio (contact angle 150.3 $\pm$ 2.1°) while other formulations were also hydrophobic (contact angles 121 to 135°). We also examined Methanol (boiling point of 148.5 °F) in the same volume that, in contrast, generated hydrophilic surface coatings with all formulations (contact angles 34 to 82 °), but most prominently with the 1: 2 at the higher concentration (contact angle 80.7 $\pm$ 2.1 °).

Among the two solvents examined, Methanol did not enable a suitable anti-fogging formulation and hence, the 1: 2 formulation in Acetone was chosen as an optimal anti-fogging solution.

The use of surface disinfectants is an integral part of maintaining and reusing PPEs. This is a sustainable and cost-effective practice that is routinely employed. A key design requirement of the anti-fogging solution is durability with these disinfectant approaches. We observed that the soap solution group consistently demonstrated complete removal of the anti-fogging solution. While this desirable as a disinfectant approach to remove all potentially hazardous aerosols on these PPEs, it would not be suitable with the applied anti-fogging solution. The removal of the anti-fogging solution with these approaches could be attributed to the surfactant nature that interferes with the surface tension of the water droplets. Another possibility is that the lipid-to-lipid interaction of the soap and the waxes may inactivate the hydrophobic nature of the coating. These remain to be investigated in future studies.

To investigate the durability of the anti-fogging coating, we generated a custom rig for accelerated surface contact testing. The major design principle for this device was to generate a PPE skin contact force. The reported PPE skin contact force to create a tight seal on the nose and chin has been reported as 45 to 91 mm of Hg (6000-12,000 N/m^2). (30-32) The force generated by our rig was a little lower at 5000 N/m^2 that approximates light skin contact force in non-fastened areas. This reflects routine surface contact during routine use of the PPEs. The integrity of the anti-fogging surfaces was relatively well maintained (contact angle 133.3 ± 1.5^o and sliding angle 23.7 ± 1.5^o). However, the durability testing noted a reduction in the optical clarity of the masks that could be attributed the wear from the contact forces. As evident in the ultrastructural analysis, the PETG surface appears to have several accretions and disruption of the uniform coated surfaces. Given these PPEs are disposable and have a limited time of use, this may not significantly impact the PPE performance. In fact, this lack of clarity from routine use may be a good marker for replacement with a simple optical interferometry device.

This study has a few limitations. First, the biological performance of this anti-fogging solution needs to be validated using *in vitro* (reconstructed human epidermis), and *in vivo* skin contact testing in animals and human studies. (40-46) Second, applying a superhydrophobic solution on the PETG clear surface may, counter to its primary objective, worsen the visibility due to the moisture coalescing into larger water beads. It is conceivable that the increasing size of these water beads may eventually reach a critical mass and cause them to slide off. This phenomenon is observed on the lotus leaves and commercial car wax coatings. Hence, a replaceable absorbent liner and a mild, mechanical vibration module to promote water droplet beading and run-off could be future design iteration in these clear PPEs. Third, a major shortcoming of using such an anti-fogging spray is its temporary nature and the need for continuous replacement throughout the lifetime of the clear PPE. Thus, a potential future possibility is to pursue nanopatterning, simulating the lotus leaf papillae for a more biocompatible, permanent anti-fogging solution.

Conclusions

In summary, this work demonstrates the utility of an anti-fogging formulation with two natural waxes that has good durability and optimal non-fouling properties. This solution appears to represent a cost-effective, durable, non-toxic approach to prevent or reduce fogging of clear PPEs such as plastic masks and face shields from condensations during humidity natural breathing and speaking.

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Conflicts of Interests

The authors declare they have no relevant conflicts of interest with this work.

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Figure Legends

Figure 1. Physical characterization of anti-fogging coating

(**A.**) CAD of the contact angle apparatus for 3D printing (**B.**) CAD of the sliding angle module for 3D printing (**C.**) 3D printed models of contact and sliding angle testing apparatus (**D.**) image of the contact angle assessment showing droplet generation and analysis. Inset shows the digital analysis of contact angel ϕ using NIH ImageJ (**E.**) Digital image analysis showing the sliding angle measurement using NIH ImageJ (**F.**) Optical clarity set-up using a diode laser and optical power meter with sensor.

Figure 2. Anti-fogging solution formulation and characterization

(**A.**) Tabular outline of the formulation depicting ratios of carnauba and beeswax in acetone and methanol used to coat PETG plastic sheets (**B.** and **C.**) Contact angle assessment of Acetone and Methanol formulations at various concentrations (**D** and **E**) Sliding angle analysis of Acetone and Methanol formulations at various concentrations (**F** and **G**) Transmission of diode laser for optical clarity assessment of Acetone and Methanol formulations at various concentrations. Data is shown as Mean and SD and is representative of atleast two independent studies. Statistical significance was determined using two-way analysis of variance (ANOVA) among different treatments using the Bonferroni's multiple comparison test ($n = 3$). Statistical significance is denoted as * $p < 0.05$, ** $p< 0.005$, and *** $p< 0.0005$.

Figure 3. Effects of Disinfection on anti-fogging coating integrity

(**A.**) Contact angle assessment after various disinfection procedures (**B.**) Sliding angle analysis after various disinfection procedures (**C.**) Optical clarity after various disinfection procedures. Data is shown as Mean and SD and is representative of atleast two independent studies. Statistical significance was determined using two-way analysis of variance (ANOVA) among different treatments using the Bonferroni's multiple comparison test ($n = 3$). Statistical significance is denoted as * $p < 0.05$, ** $p< 0.005$, *** $p< 0.0005$, and **** $p< 0.00005$.

Figure 4. Durabilty testing of anti-fogging coating following contact testing

(**A.**) CAD of contact testing apparatus used for wear testing analysis (**B**.) 3D printed model of the assembled contact testing apparatus (**C.**) Outline of the eminent forces determining the contact force on PETG surfaces. The contact angle (**D.**), sliding angle (**E.**), and optical clarity (**F**.) analysis before and after contact testing (**G.**) Scanning electron microscopy images of the uncoated and coated surfaces before and after contact testing. Data is shown as Mean and SD and is representative of atleast two independent studies. Statistical significance was determined using Student T test ($n = 3$). Statistical significance is denoted as * $p < 0.05$.

Figure 1

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Fig. 1

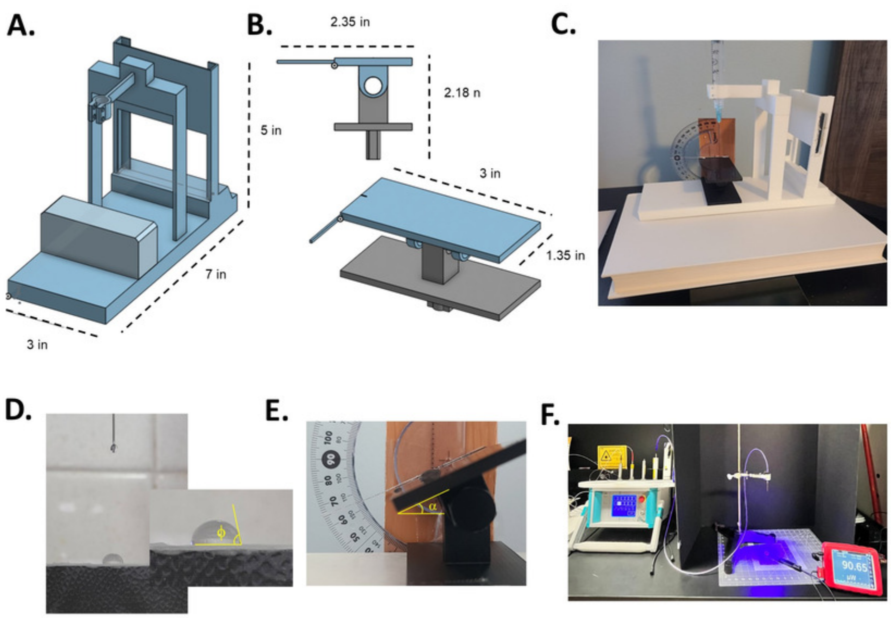

Figure 2

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Fig. 2

A.

Ratio	Carnauba (g)	Beeswax (g)	Acetone/Methanol (ml)
1:1	0.4375	0.4375	25
1:1.5	0.35	0.525	25
1:2	0.33	0.67	25
1:2 (HC)	0.417	0.833	25

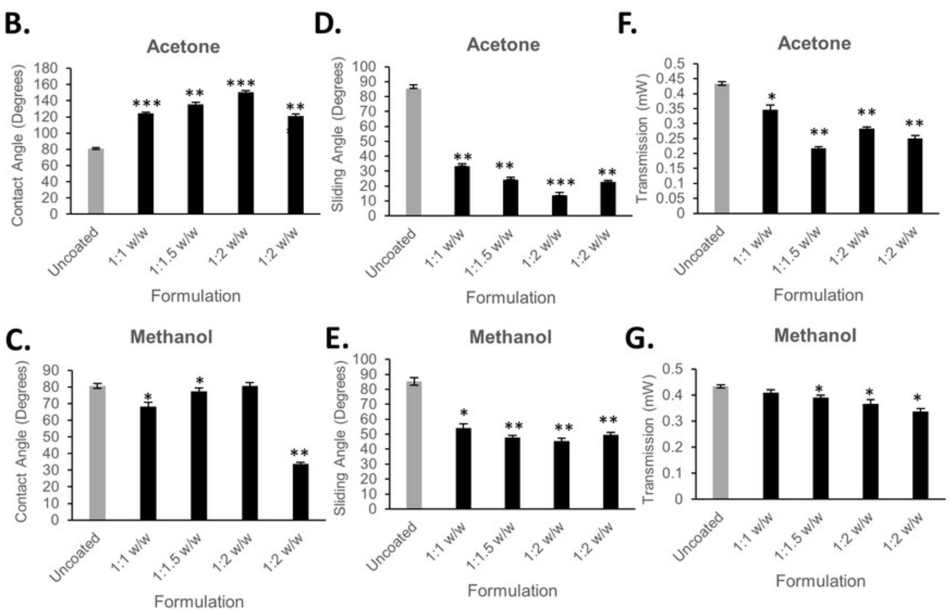

Figure 3

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Fig. 3

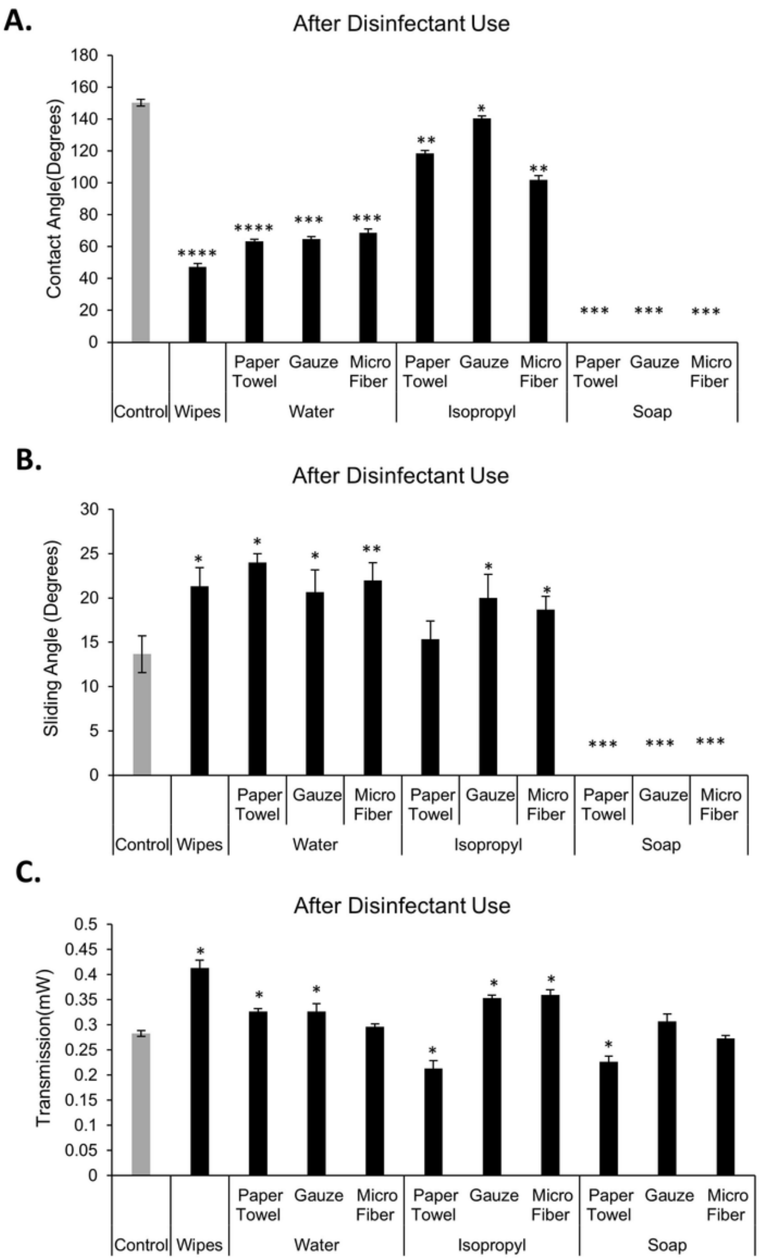
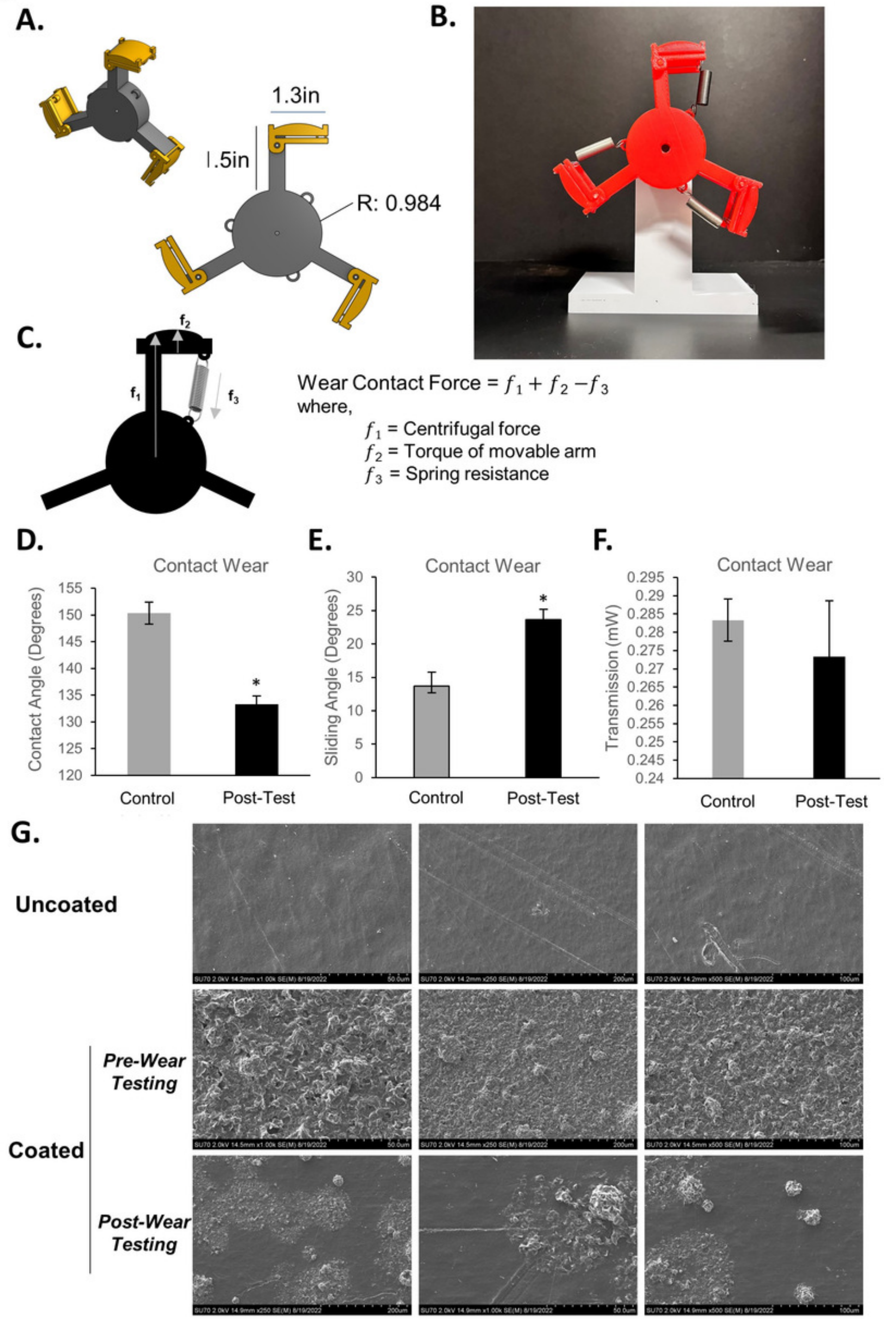

A.
After Disinfectant Use
Contact Angle(Degrees)
180
160
140
120
100
80
60
40
20
0
****
****
***
***
**
*
**
***
***
***
Paper Towel
Gauze
Micro Fiber
Control
Wipes
Water
Isopropyl
Soap
B.
After Disinfectant Use
Sliding Angle (Degrees)
30
25
20
15
10
5
0
*
*
*
**
*
*
***
***
***
Paper Towel
Gauze
Micro Fiber
Control
Wipes
Water
Isopropyl
Soap
C.
After Disinfectant Use
Transmission(mW)
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
*
*
*
*
*
*
*
Paper Towel
Gauze
Micro Fiber
Control
Wipes
Water
Isopropyl
Soap

Figure 4

Figure 4. Durability testing of anti-fogging coating following contact testing

(**A.**) CAD of contact testing apparatus used for wear testing analysis (**B**.) 3D printed model of the assembled contact testing apparatus (**C.**) Outline of the eminent forces determining the contact force on PETG surfaces. The contact angle (**D.**), sliding angle (**E.**), and optical clarity (**F**.) analysis before and after contact testing (**G.**) Scanning electron microscopy images of the uncoated and coated surfaces before and after contact testing. Data are shown as Mean and SD and representative of at least two independent studies. Statistical significance was determined using the Students' T-test (n = 3). Statistical significance is denoted as * $p < 0.05$.

Fig. 4


A.
1.3in
1.5in
R: 0.984
B.
C.
f1
f2
f3
Wear Contact Force = $f_1 + f_2 - f_3$
where,
f_1 = Centrifugal force
f_2 = Torque of movable arm
f_3 = Spring resistance
D.
Contact Wear
Contact Angle (Degrees)
Control
Post-Test
E.
Contact Wear
Sliding Angle (Degrees)
Control
Post-Test
F.
Contact Wear
Transmission (mW)
Control
Post-Test
G.
Uncoated
Coated
Pre-Wear Testing
Post-Wear Testing