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Effects of multiple wetting incidents, shear and sliding friction on lubricant stability in SLIPS

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ABSTRACT

Surface icing almost invariably derives from the precursory step of liquid water encountering the surface. Thus, slippery liquid infused porous surfaces, SLIPS, must possess steady wetting durability, and lubricant stability to function as a reliable hydro−/icephobic surface design especially in outdoor applications. Additionally, they should maintain their phobic performance under shear forces, and possess low sliding friction to act as a slippery, multirepellent surfaces. These characteristics are needed in variable applications ranging from moving and rotating blades to steady surfaces, operating in altering climate conditions. More profound durability testing is needed to examine the loss of surface functionality when the lubricant is depleted from the structure via various routes. In addition, the durability tests should be designed to serve the application-related purposes and thus, to reveal performance differences between slippery surfaces for further analysis and targeted end-use development. Here, we tested the wetting durability and stability of SLIPS with multicycle Wilhelmy plate by dipping the surfaces multiple times in water bath. Additionally, we examined the effects of centrifugal and friction-based shear stress to investigate the lubricant depletion from the structure. Tests that measure the durability and the stability of SLIPS designs are in great need in further developing functional slippery surfaces for real outdoor application coatings which encounter environmental stresses, e.g., wetting and icing. Acknowledging the material differences under specific stresses will guide designing the slippery surfaces towards more specific and functionable end-use applications.

1. Introduction

Slippery liquid infused porous surfaces, SLIPS, possess excellent droplet mobility and exceptional repellency towards various of liquids (Wong et al., 2011; Leslie et al., 2014; Daniel et al., 2013a). This repellent nature is seen even in cold environments as SLIPS can hold off frost, condensation, inhibit ice nucleation and have icephobic characteristics such as extremely low ice adhesion (Niemelä-Anttonen et al., 2018a; Subramanyam et al., 2013; Irajizad et al., 2016; Donadei et al., 2021; Juuti et al., 2017; Anand et al., 2012; Wilson et al., 2013; Jin et al., 2018). These multifunctional properties of the SLIPS technology arise from the impregnated lubricant and the porous solid material which together comprise the slippery surface. The two-component surface design differs greatly from solid surfaces. When in contact with immiscible material in gaseous atmosphere SLIPS form a four-phase-contact-line (Smith et al., 2013) with the solid, the lubricant, the surrounding gas, and the immiscible material. This leads in unique characteristics when any of these four is changed highlighting the complexity of slippery liquid infused porous surfaces.

Even though the lubricant enables many prominent properties, it also causes some noteworthy issues. That is, the main hindrance of SLIPS is related to their durability and lubricant stability under environmental stresses (Niemelä-Anttonen et al., 2018a; Liu et al., 2016; Howell et al., 2015a; Coady et al., 2018). Altering climatic conditions, such as rain, wind and temperature changes, can cause the lubricant to be diminished from the solid structure. This depletion may occur from the topmost lubricant layer by contacting water droplet cloaking and wetting ridge formation (Anand et al., 2012; Smith et al., 2013; Schellenberger et al., 2015; Sett et al., 2017; Preston et al., 2017), under liquid stream or flow (Baumli et al., 2021), evaporation (Niemelä-Anttonen et al., 2018a; Anand et al., 2012; Zhang et al., 2014), shearing/dynamic forces (Baumli et al., 2021) and/or by gravitational drainage (Smith et al., 2013). These depletion routes alter the characteristics and the functionality of the slippery surface, compromising the repellent and

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slippery nature. As the lubricant is lost from the structure, the porous solid material is exposed leading to droplet impinging (Preston et al., 2017), and in the case of freezing temperatures, increase of ice adhesion force (Niemelä-Anttonen et al., 2018a). Also, the behavior of the porous solid under environmental stress contributes significantly to the longevity of SLIPS.

There are various of durability tests for coatings and modified surfaces, however, the end-use application should drive the durability testing, preferably indicating the real performance differences between the surface designs. The durability of various SLIPS has previously been examined, e.g., by shear stress under water/air flow (Wexler et al., 2015; Howell et al., 2015b), where the flow consumes the lubricant from the SLIPS surface, mimicking the running water flow effect. Alternatively, the surfaces have been placed into a water bath with a constant stir (Wei et al., 2016) to mimic similar water erosion effect. Other ways to examine the overall durability include evaporation studies (Niemelä-Anttonen et al., 2018a; Wei et al., 2016), mechanical durability and abrasion (Tenjimbayashi et al., 2016), and shear stability tests, e.g., by centrifugal spinning (Wei et al., 2016; Kim et al., 2013). Examining SLIPS under mobile interfaces, such as force tensiometer studies in water/air, provides vital aspects in examining the lubricant depletion and it's effects on wettability performance (Peppou-Chapman and Neto, 2018; Peppou-Chapman and Neto, 2021).

The surfaces engineered for cold environments must possess required key properties to function on top a specific application for a certain lifespan. Due to this, we have previously examined SLIPS wetting characteristics by sessile drop methods in terms of lubricant evaporation, freeze-thawing performance, and icephobic performance under high velocity impacting supercooled water droplets (Niemelä-Anttonen et al., 2018a). Here, investigating larger sample areas yields comparative methodology to examine wetting but more importantly to evaluate the lubricant depletion under full water immersion with multiple wetting incidents. These long-term wetting characteristics can play a major role in hydro- and icephobic applications as the SLIPS must endure multiple wetting cycles without losing their phobic performances. To evaluate the wetting endurance and durability, we performed multicycle Wilhelmy plate to analyze the forced wetting by immersing the samples to cold water. Moreover, the lubricant stability is a fundamental feature in many applications, especially in rotating devices, such as in drones and windmill blades. Thus, to understand the porous solid material and lubricant interoperability, we examined the lubricant stability under shear force created by centrifugal spinning and also by sliding friction. Most importantly, the overall durability assessment highlights the performance differences which are to be mapped for specific end-use coating/surface modification development.

2. Methods and materials studied

SLIPS are comprised of the porous solid material and the lubricating agent. In present study, the used lubricants were perfluorinated Krytox 103 high performance lubricant with viscosity of 80 cSt (DuPont, USA), and silicone oil with viscosity of 50 cSt (Sigma-Aldrich, USA). The porous solid materials were nonwoven nanofibrous membranes of polytetrafluoroethylene, PTFE, and polypropylene, PP, (Sterlitech Corporation, USA), which were used in as-received conditions. The average pore size for both polymeric porous solids was 0.2 μm, as given by the supplier. The thickness of these porous solid membranes ranged between 25 and 110 μm according to the specifications of the given supplier. The SLIPS samples were prepared as depicted in our earlier work (Niemelä-Anttonen et al., 2018a): the porous solid membranes were impregnated with lubricants on top of fully oil-immersed filter papers. This method enables the membrane to wet and wick the lubricant passively into the pores with mere capillary forces. For each test, four parallel samples were prepared and examined.

2.1. Wetting endurance test with tensiometer

For multicycle Wilhelmy plate method, the porous membranes were attached to thin coverslips (24 × 40 mm, Menzel-Gläzer, DE) by double sided tape and the edges of the membranes were sealed with water-resistant glue (Henkel, Loctite®, DE). The tensiometer (KSV Sigma 70, KSV Instruments, FI) moved the sample plate with a speed of 12 mm/min into the water bath with immersion depth of 10 mm. The water used in the experiments was ultra-high purity water (MilliQ, Millipore, USA) with resistivity under 18 MΩ•cm, and surface tension of 71.8 ± 0.4 $\mathrm{mN \cdot m^{-1}}$. The lubricant loss was examined by weighting the samples before and after the wetting endurance cycles, with high precision scale with the precision of 0.1 mg.

2.2. Lubricant stability test with centrifugal spinning

The lubricant stability was examined by centrifugally spinning the samples in a centrifuge (AK15, Sigma, DE) at 2000 rpm for 30 min. The membranes were attached to microscope slides with double sided tape, immersed with the lubricants and placed into 50 mL centrifuge tubes. As in the wetting endurance tests, the lubricant depletion was observed with the high precision scale.

2.3. Friction measurements

The friction and the sliding properties of the SLIPS were examined with friction tester (FX-7000, Oakland Instrument, USA) connected to a load cell with a minimum reading of 0.1 g. The sample surfaces were attached under the sled and pulled with a constant speed on top a silicone paper (siliconized yellow glassine, UPM Raflatac, FI) adapting the ASTM D1894. This liner material was chosen for its good availability, slick and smooth surface, and inert nature of silicone as a material. The samples were attached into 30 × 30 mm sled with double sided tape. An extra weight was placed on top of the sled bringing the net weight of the sled to 99.7 g.

2.4. Microscopy and characterization

The topographies of the membrane surfaces were examined by optical profilometry (Alicona Infinite Focus G5, AT) with 20× objective magnification. The field area of the measurements was 0.8 mm × 0.8 mm in the xy-plane. The surface structures were visualized by field-emission scanning electron microscope (Zeiss ULTRAplus, Carl Zeiss SMT AG, DE).

3. Results and discussion

3.1. Wetting durability by multicycle Wilhelmy plate method

The multicycle Wilhelmy plate tests were carried out in order to get more profound information of the performance of SLIPS under multiple wetting incidents in respect to water contact angles. Apart from more often used droplet-based optical goniometer, this method utilizes larger studied area, pinpointing possible surface defects in micro and macro scale; shows the surface resistance under forced wetting condition, indicating the stability of the lubricant in the porous solid; and also yields information on the basic wetting properties (Moghaddam et al., 2016; Tretinnikov and Ikada, 1994; Volpe, and della, Siboni S., 2018). Investigating these parameters in SLIPS, the lubricant lockage and stability by porous solid capillaries can be evaluated. Thus, we tested the wetting durability by dipping the samples into water bath for 20 cycles in room temperature (RT) and in the temperature of +5 °C. These temperature points were chosen to investigate the temperature-dependent water-surface interactions. Firstly, water approaches the densest form around +5 °C slowing and changing the molecular structures providing interesting comparison to room temperature test points.

Secondly, materials behave differently in cold environment possibly bringing out crucial performance differences for application-driven surface development. The tested samples consisted of (super)hydrophobic polymer membranes and SLIPS comprised from these porous membranes by impregnating them with silicone and perfluorinated oils. Fig. 1 illustrates the wetting events for one wetting cycle performed with Wilhelmy plate.

The (super)hydrophobic polymeric porous solids of PP and PTFE showed dimensional stability as no changes were observed in liquid uptake, nor there were any changes in the probe force in wetting curves, as illustrated in Fig. 2. The hysteresis, the difference in between the advancing contact angle (ACA) and the receding contact angle (RCA), is greater in plain membranes than in SLIPS, a trend also seen with optical goniometer due to Cassie-Baxter wetting state caused by the lubricant (Niemelä-Anttonen et al., 2018a). Measured by Wilhelmy plate method, the CAs in RT were 153° and 135°, for PTFE and PP, respectively. These distinctive water contact angle values arise from the chemical nature of the polymer, and from their porosity characteristics. The CA is higher due to the (super)hydrophobic nature of the solid, enhanced by their porosity which traps air into the pores.

After the lubricant impregnation, the CAs diminished due to more Wenzel-like lubricant / water interaction (Niemelä-Anttonen et al., 2018a). In the SLIPS, the pores are filled with lubricating liquid, creating extremely smooth and uniform interface, lowering the contact angle of the water. The silicone oil impregnated SLIPS showed slightly decreased CAs, 105° in both cases (PTFE and PP), whereas for perfluorinated oil the SLIPS showed 118° CA in PTFE, and 113° in PP. Due to the presence of the lubricant, the contact angles decrease with a causal connection between the four-phase-contact-line, where each component changes the physio-chemical relations, resulting in unique contact points and thus, unique contact angles and wetting behavior for each SLIPS design (Niemelä-Anttonen et al., 2018a; Juuti et al., 2017; Baumli et al., 2019).

Yet, in the SLIPS the lubricant depletion is detected from the slight change in the force within the cycles as the wetting curves slightly changed with increasing cycle number. The depletion was confirmed by measuring the surface tension of the water bath after the wetting cycles. The surface tension of the water declined in each test due to the presence of lubricant, as seen from Supplementary Information Table S1. Table 1 presents the lubricant loss percentage in weight percentage for the SLIPS with the prominent effect of the bath temperature: the lubricant is depleted more in cold water than in RT. We argue that the decrease in temperature causes the polymeric membranes to shrink pushing the lubricant away from the pores leading to greater lubricant loss in the lower temperature (Jiang et al., 2019; Yao and Nagarajan, 2004; Shashoua, 2014; Shelesh-Nezhad and Taghizadeh, 2007). This effect was observed in all the SLIPS tested in +5 °C, also in the shear force durability test, described in the next section.

Overall, the plain membranes maintain stable wettability without changing over the 20 cycles in both temperatures. This indicates that the 0.2 μm pore size was able to withstand water penetration over multiple forced wetting incidents and thus, keep the interconnected porosity filled with air in both polymer materials, PP and PTFE. The SLIPS performed equally steadily within the test setup, as no appreciable changes were seen in the CAs. Still, some minor amount of lubricant was depleted from the structure as depicted in Table 1. The physio-chemical material differences between PTFE and PP might result in different depletion amount throughout the tests. These depletion observations are in line with other studies (Peppou-Chapman and Neto, 2018; Peppou-Chapman and Neto, 2021), showcasing the challenge of designing a porous solid porosity and capillary structure which would hold the lubricant within even under forced wetting states.

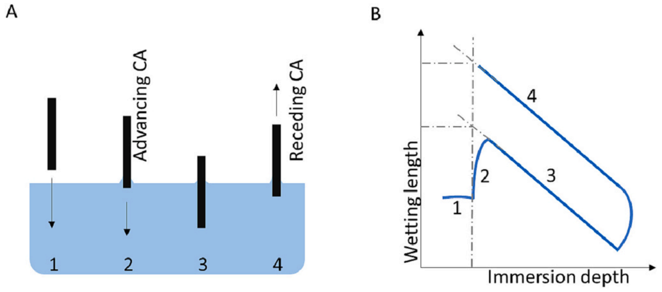

Fig. 1. The Wilhelmy plate method by force tensiometer yields values from advancing contact angle as the sample is lowered to water bath, and also receding contact angle as the sample is pulled from the water.

3.2. *Lubricant stability under centrifugal shearing force*

To examine the lubricant stability differences under shearing forces, we studied the lubricant depletion from the SLIPS by centrifuging the samples in two different temperatures, RT and + 5 °C. The shear stability tests with centrifugal spinning (Wei et al., 2016; Kim et al., 2013) have previously been reported only for short period of time. However, we examined the depletion rates by exposing the surfaces to 2000 rpm for 30 min in order to bring out the differences of the tested SLIPS stemming from the porosity/capillary effect, the chemical properties of the solid-liquid material pairs, and the lubricant characteristics. Table 1 illustrates how the lubricant depletion increased in lower temperature of +5 °C compared to RT. Similarly to multicycle Wilhelmy plate, performed in the same temperatures, the depletion was greater in the cold environment than in RT presumably due to the polymer matrix shrinkage as speculated earlier. This material specific thermal shrinkage (Shashoua, 2014) characteristics might be forcing the lubricant to migrate towards the SLIPS surface providing an excessive and unstable lubricant amount on top. Even though PTFE is known for its excellent insulating properties (Blumm et al., 2010), in SLIPS where a porous polymeric solid is incorporated with lubricant, one of the limiting factors might arise from the temperature-dependent dimensional stability of the used polymers. For both of the porous solids, PTFE and PP, the average pore size is the same 200 nm as given by the supplier and as characterized with electron microscopy in the following chapter. The finer fibrous PTFE (Niemelä-Anttonen et al., 2018a) lost greater amounts of the impregnated lubricant from the structure in RT, and in +5 °C. Additionally, the lubricant chemistry was shown to contribute the depletion amount. Particularly in the +5 °C, the loss of the silicone oil was lesser than that of the perfluorinated oil. This trend is seen on both slippery designs, in PP-SLIPS and in PTFE-SLIPS. Both of the used lubricating oils possess similar viscosity range of 50 to 80 cSt in normal room temperature 20–22 °C, promoting the properties of lubricant chemistry. For silicone oil, polydimethylsiloxane, the linear chain of siloxane repeating units (–Si–O) provides the special property of molecular and materialistic flexibility whilst being a strong bond (Eduok et al., 2017). Likewise, the Krytox 103 fluorinated oil, polytetrafluoroethylene, is a low molecular weight fluorine end-capped homopolymer with strong (–C–F) bonds resulting inertiveness (Dhanumalayan and Joshi, 2018). Additional difference is the surface tension of these lubricants, for Krytox 103 recorded values are 17.1 mN/m, and for silicone oil 21.1 mN/m (Daniel et al., 2013b; Fujimatsu et al., 2003; Li et al., 2012). The observed differences in lubricant depletion might stem from differences in chemistry but most notably the surface tension and the viscosities play a role. The importance of profitable chemical compatibility between the porous solid material and the lubricating oil is seen to delay the lubricant depletion (Peppou-Chapman and Neto, 2018). Similarly, the differences of capillary and viscous forces are seen to attribute to dynamic wetting experiments (Mohammad Karim and Kavehpour, 2018).

3.3. *Slippery and friction properties*

Frictional properties in SLIPS play an important role in repelling

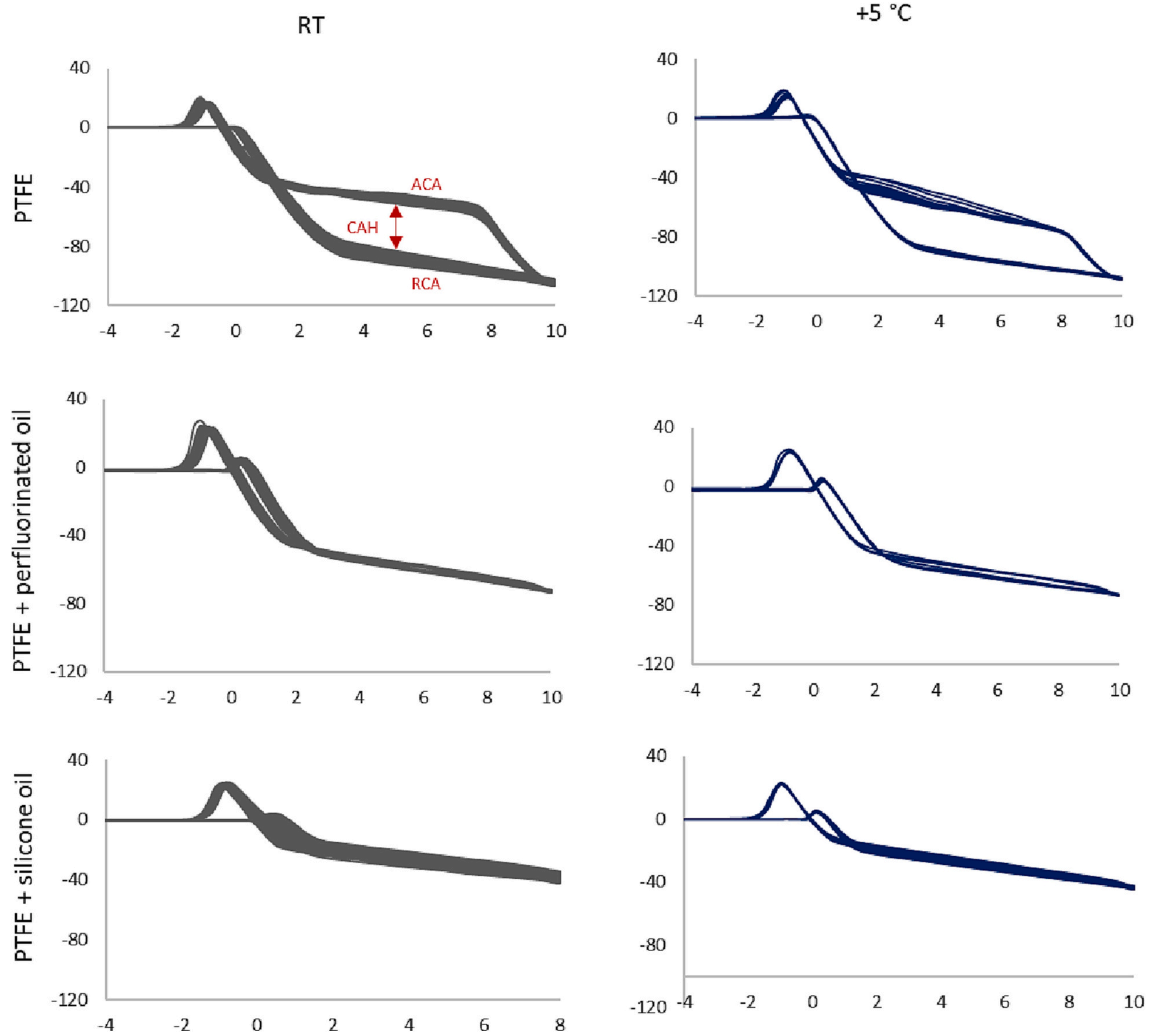

Fig. 2. The graphs illustrate the wetting performance for PTFE in RT and in +5 °C for 20 wetting cycles, similar trends in ACA and RCA are seen with PP in Supporting information S2. The hysteresis is greater in unfilled porous solids, however, the SLIPS endure multiple forced wetting cycles without changes in CA. The curves show force with the sample perimeter, F/P (mN·m^{-1}) on y-axis; versus immersion depth, h (mm) on x-axis.

Table 1
The lubricant loss of the SLIPS measured with: Wilhelmy plate after 20 cycles; and centrifugal shear after 2000 rpm / 30 min.

LUBRICANT LOSS in SLIPS				
	Wilhelmy plate, 20 cycles		Centrifugal shear, 2000 rpm, 30 min	
	RT	+5 °C	RT	+5 °C
PTFE + silicone oil	13,3%	16,5%	90,3%	91,8%
PTFE + perfluorinated oil	10,2%	14,9%	89,5%	93,1%
PP + silicone oil	1,5%	4,0%	53,5%	57,8%
PP + perfluorinated oil	0,5%	1,0%	55,8%	79,5%

immiscible materials, such as ice, and understanding the effects of material selection in developing SLIPS designs. Here, the plain membranes possess the same nominal porosity of 0.2 µm, however, their structure and material properties play a major role in the tests. Arguably, the different surface roughness patterns affect the real area of contact in the friction test, as to the polymer bulk properties (Quaglini et al., 2009). In SLIPS, the real area of contact is expected to be higher due to the lubricating layer which creates uniform layer in between the two surfaces: the sample and the test surface. Even though the contact area is supposed to be grater, the friction commonly declines after introducing the lubricating agent in between two solids. This is especially seen in the case of PTFE where the kinetic friction coefficient diminished from 3.6 to 0.4 after impregnation, as observed from the Fig. 3. Surprisingly, same kind of decrease does not occur in the PP.

The PP presented extremely steady friction behavior towards the test surface, with and without impregnation. In this rougher fibrous membrane, the friction coefficients are remarkably similar for the plain membrane and for the PP-SLIPS. Interestingly, the lubricant type seemed not to have a significant impact on the friction properties in this test setup since the perfluorinated and the silicone oil impregnated SLIPS behaved similarly. FESEM images show the surfaces to undergo not only elastic deformation but also plastic deformation, leading to fractures of the structure in both membranes, as depicted in Fig. 3C.

The nominal friction of the material stems from the molecular level properties, but also from the material roughness and its deformations. Increase in friction is arguably compromising the desired slipperiness

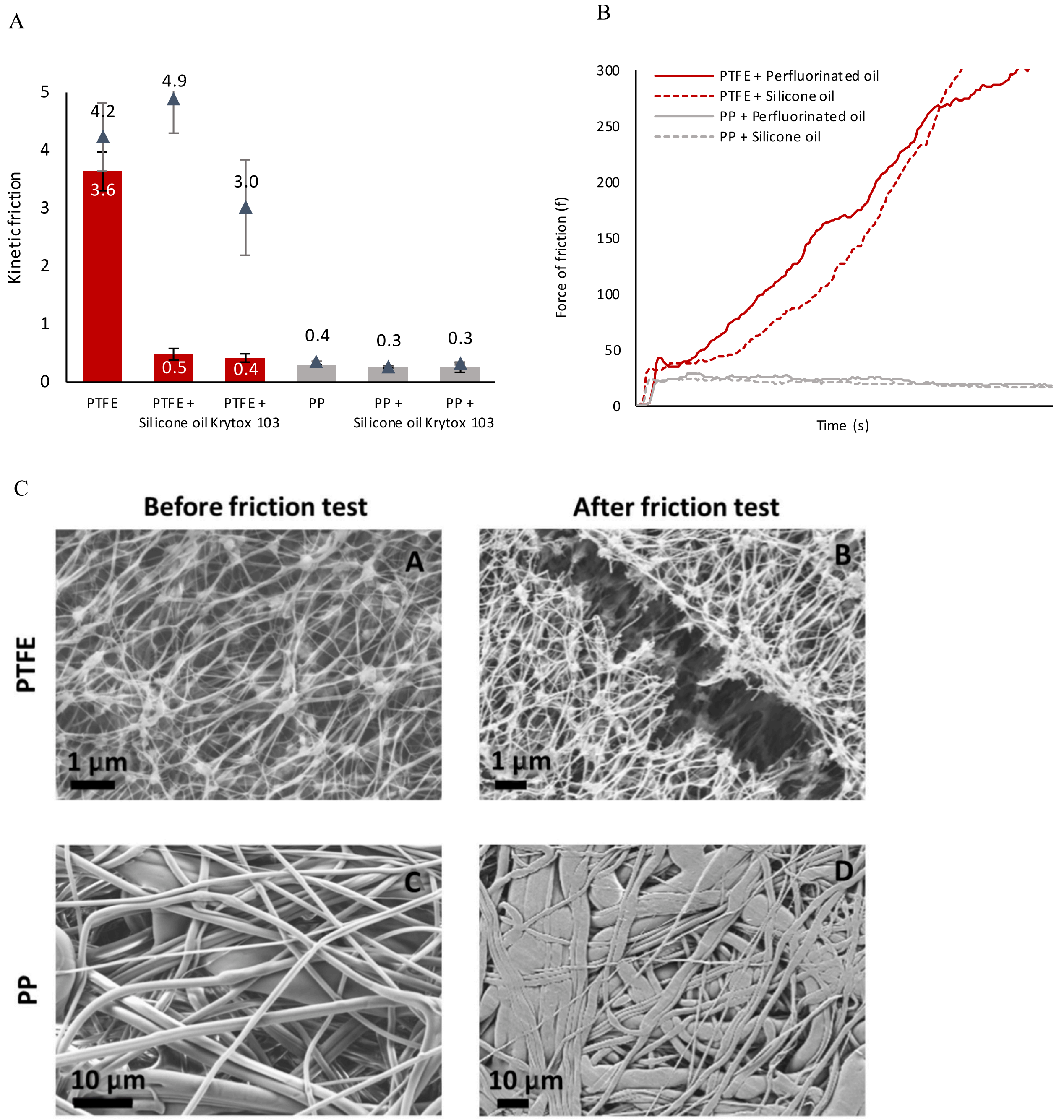

Fig. 3. A.) The friction properties of PTFE and PP with and without lubricant impregnation. The bars depict the kinetic friction coefficient, and triangular dots represent the maximum kinetic friction coefficient value. Maximum kinetic friction coefficient (FC) represents the highest recorded value of the kinetic friction. B.) The friction increment in SLIPS under constant force. The force of friction (f) is in y-axis and time (s) the datapoints recorded is in x-axis. C.) The SEM images before and after the friction test for PP and PTFE membranes depicting the material ruptures that occur during the test.

and might result in droplet impinging and increase in ice adhesion in sub-zero temperatures. Frictional shear also damaged the porous solid material resulting in deteriorating the SLIPS by tearing the material, flattening the porosity, and thus impairing the lubricant stability within, as illustrated in Fig. 3.

If the porous solid capillary effect is deformed, it can lead in increased lubricant depletion. Additionally, the flattened surfaces might push the lubricant from the inner structure towards the surface increasing lubricant loss. Studies indicate the excess lubricant film leading to unreliable test results or even to rapid failure of SLIPS properties due to high level cloaking and wetting ridge formation (Niemelä-Anttonen et al., 2018a; Schellenberger et al., 2015; Mohammad Karim and Kavehpour, 2018).

4. Conclusions

In our previous publications, we showed that SLIPS can exhibit excellent characteristics towards low ice adhesion, resisting multiple freezing events and possessing promising wetting behavior (Niemelä-Anttonen et al., 2018a; Juuti et al., 2017; Niemelä-Anttonen et al., 2018b). From the icephobic perspective, the most prominent material pair studied was 0.2 µm pore size PTFE, impregnated with 50 cSt viscosity silicone oil, yielding extremely low ice adhesion of 8 kPa (Niemelä-Anttonen et al., 2018a). Here, we focused more on the durability of selected SLIPS and investigated the nominal differences of these designs by examining: a) wetting durability test with multicycle Wilhelmy plate; b) lubricant stability study with centrifugal shear, and c) friction properties examination by sledge test.

Even though, the PTFE derived SLIPS obtained record low ice adhesion under icing wind tunnel accreted ice (Niemelä-Anttonen et al., 2018a), this slippery design showed unstable behavior in cold temperature durability tests. PTFE-SLIPS had grater lubricant loss than PP-SLIPS in both, multicycle Wilhelmy plate and centrifugal shear tests. In Wilhelmy method, the lubricant chemistry played minor role in depletion, however, under centrifugal shear the silicone oil seemed to be more stable within the porous structures. From the solid materials perspective, the porosity and the roughness are important factors in determining other notable performance differences, as seen with the friction analysis. The more fibrous and rougher PP outperformed PTFE in the used test setup, a profitable characteristic with no interdependence for neither silicone nor perfluorinated lubricant. From materials science perspective, more analysis is needed for SLIPS from the material performance point of view. That is, understanding the material compatibility, in terms of thermal expansion of solid-liquid pairs, as to suitable porosity and lubricant chemistry to hold the physical properties under specific climatical conditions, should be well acknowledged for next level SLIPS design.

We have shown that end-use conditions and requirements of specific application are extremely important when selecting the most prominent SLIPS-solution. Noteworthily, these SLIPS are well established in scientific literature since their initial appearance in 2011 (Wong et al., 2011). Due to this, by showcasing their varying performance, not only under freezing temperatures but also under more elementary durability test setups, their material differences are distinguished. This highlights the application driven necessity to understand the end-use conditions, especially the environmental stresses the coating will most likely encounter. From surface engineering point of view, there might not be an omnipotent slippery design to outperform under vast array of stresses and climatical conditions.

More well-constructed and application-driven tests are needed to examine the SLIPS properties, to compare their functionality, and to develop their durability to meet the real outdoor application requirements for slippery coatings.

CRediT authorship contribution statement

Henna Niemelä: Conceptualization, Methodology, Writing – original draft, Writing – review & editing. **Mikko Tuominen:** Methodology, Supervision, Writing – review & editing. **Heli Koivuluoto:** Supervision, Writing – review & editing. **Petri Vuoristo:** Supervision, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

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