Controllable Friction of Green Ionic Liquids via Environmental Humidity

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Intelligent control of friction is an attractive but challenging topic. In this work, it is investigated if it would be possible to adjust friction in a lubricated contact by controlling environmental humidity. By exploiting the ability to adjust the environmental humidity by various saturated salt solutions, friction behavior of contacts lubricated with Choline L-Proline ([Cho][Pro]) is modulated in a wide range of relative humidity (RH). The friction increases when the environmental humidity is increased and decreases when water is partially evaporated to a lower RH. It is thus possible to control friction by environmental humidity. The addition of water in ionic liquids (ILs) causes a decrease in viscosity, but as the tests are calculated to be performed in boundary lubrication the viscosity change is not the main factor for the change in friction. The friction sensitivity of RH can be explained by the effect of adhesion on the water uptake from humid air by [Cho][Pro]. Furthermore, the reversible changes of H-bond types determined by the water content could be another explanation to the altered friction.

1. Introduction

Today the demand for intelligent control of tribological interactions is strongly increasing in various applications. We often strive to minimize friction but there are also many situations where high friction is desirable. In some cases, something in between is attractive, i.e., optimum friction. Driven by the broad application prospects, many controllable friction devices regulated with external stimuli have been designed and fabricated. Some examples of external stimuli are solvents,[1–7] pH,[8–10] temperature,[11–13] electric potential,[14–16] light,[17–19] and magnetic fields.[20–22] When external stimuli are imposed on such smart materials, the macroscopic physicochemical properties of the materials are dramatically changed. For example, when a UV light is applied to a liquid-crystal coating, 3D fingerprint structures can be induced and modulated.[4]

In various fields of applications, tribosystems are required to work without damage under changing environmental conditions, while in many outdoor applications temperature and relative humidity (RH) can undergo large variations because of geographical and seasonal changes. The effect of humidity on tribological performance, however, seems not to be given the same importance as other environmental factors (e.g., temperature and atmosphere).[23] Water, in the form of moisture for unlubricated contacts or water absorbed by a lubricant from the environment, can have a significant effect on the tribological performance, but the influence of humidity on friction is complicated and there is no universal law. Even for dry lubrication, the addition of water can have different effects. For example, absorbed water in high RH is of benefit to be able to capture moisture when used as a lubricant. In various fields of applications, tribosystems are required to work without damage under changing environmental conditions, while in many outdoor applications temperature and relative humidity (RH) can undergo large variations because of geographical and seasonal changes. The effect of humidity on tribological performance, however, seems not to be given the same importance as other environmental factors (e.g., temperature and atmosphere).[23] Water, in the form of moisture for unlubricated contacts or water absorbed by a lubricant from the environment, can have a significant effect on the tribological performance, but the influence of humidity on friction is complicated and there is no universal law. Even for dry lubrication, the addition of water can have different effects. For example, absorbed water in high RH is of benefit to be able to capture moisture when used as a lubricant.

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In this work, we set out to investigate if it would be possible to adjust friction by controlling the environmental humidity in a lubricated contact.

2. Results and Discussion

Before getting a basic understanding of the role of humidity in controlling friction, the water adsorption kinetics at different RHs should be studied. Björling et al. studied the water adsorption kinetics of [Cho][Pro] at one specific humidity (15% RH). Herein, we studied the absorbed moisture in [Cho][Pro] at different environmental humidities at 25 °C (Figure 1). The various RHs were achieved by the prepared saturated salt solution, listed in Table 1. The ionic nature of the ILs essentially makes them hygroscopic, and complete drying of ILs is extremely difficult. Before the adsorption kinetic test, [Cho][Pro] was heated in a vacuum oven at 70 °C for 2 h to ensure that the water content was lower than 0.1 wt%. As shown in Figure 1, even in the case of the lowest RH (8%), the absorbed water content in [Cho][Pro] reached 8.9% after being exposed to humid air for approximately 3 h. Thus, the adsorption of moisture cannot be neglected during practical application of [Cho][Pro]. On the other side, not only is it sensitive to moisture, but also a large range of water content can be achieved through altering the environmental humidity.

After the water adsorption kinetics of [Cho][Pro] was understood, the possibility to control friction through environmental humidity was investigated, as shown in Figure 2a. After the initial turns, stable friction values were observed for the tests in the RH of 8%, 33%, and 54%. The friction coefficient reached a steady state after 10 min in 75% RH. Unfortunately, the friction kept increasing continuously when the RH reached 94%. The changing trend in the measured friction coefficient values in the presence of different environmental humidities respectively was obvious. Generally, the higher the RH, the higher is the friction. In the presence of humid air, taking the average value over the last 5 min test (shown in Figure 2b), 8%, 33%, 54%, and 75% RH exhibited mean friction coefficient values of 0.083, 0.110, 0.134, and 0.171, respectively.

As absorbed moisture is the major substance with significant volatility, the water content after friction test can be deduced by the increased water weight. The water content was found to be 3.2%, 8.4%, 17.3%, 23.6%, 34.5% respectively. Herein, the water adsorption rate is faster than the results in Figure 1, because of sliding motion. The sliding motion, which induced shear and heat inside the fluid film, increases the absorbing rate or evaporating rate, and that explains why equilibrium state was achieved far quicker than shown in Figure 1.

To further study the possibility of online friction control through humidity, friction coefficient behaviors with both increasing and decreasing humidity were investigated. Before friction test, [Cho][Pro] was heated in a vacuum oven at 70 °C for 2 h to ensure that the water content was lower than 0.1 wt%. Two different environmental humidity conditions were created in the chamber: 1) lower humidity, lab air with approximately 20% RH was introduced to the chamber, and

![Figure 1. Water adsorption kinetics at 25 °C and different RHs.](image1)

![Figure 2. a) Friction coefficient at different RHs; b) mean friction coefficient over the last 5 min.](image2)

| Saturated salt solution | Targeted RH (%) at 25 °C |
|-------------------------|--------------------------|
| Potassium hydroxide     | 8                        |
| Magnesium chloride      | 33                       |
| Sodium dichromate       | 54                       |
| Sodium chloride         | 75                       |
| Potassium nitrate       | 94                       |
2) higher humidity, saturated sodium dichromate solution was inserted into the closed chamber to create 54% RH. As shown in Figure 3, it is obvious to see that the changing environmental humidity can greatly modulate the friction.

First, [Cho][Pro] was added between a ball and disc and absorbed moisture in the unclosed chamber (20% RH) for 60 min. After that, saturated sodium dichromate solution was placed in the closed chamber to create a higher RH. The adjustable friction behavior after that humidity was switched from 20% to 54% is shown in Figure 3a. The increase in friction lasted approximately 1 h and then a relatively stable friction coefficient was observed. Secondly, to investigate the influence of decreased humidity, [Cho][Pro] that had absorbed moisture at 54% RH for 60 min was used as an initial lubricant. Then to reduce the environmental humidity, the chamber of the SRV friction test rig was opened to ensure that the RH was approximately 20%, i.e., same as in the rest of the lab. As shown in Figure 3b, in the first half hour, the friction was at a relatively stable level, and then it started to decrease. The slow reduction may be caused by the low evaporating rate of water in ILs.

The absorbed water interacts with ILs by forming hydrogen bonds, which can modulate the ion–ion interactions and thus modify the organization of the ions. In the structure of the [Cho][Pro] molecule, carboxylate can act as a hydrogen bond acceptor, while the choline hydroxyl group can be both a hydrogen bond donor and acceptor; other H bond active groups can be found in the side chains of polar amino acids (e.g., serine, glutamic acid). These characteristics lead to the conclusion that [Cho][Pro] has the ability to absorb plenty of water, and thus it can be used to modulate the friction in a wide range of environmental humidity.

The reaction taking place within the contact area can play a central role in tribological behavior. Our previous work studied the influence of CO₂ adsorption on [Cho][Pro] and the formation of carbamate groups that strengthen the interfacial adhesion between ILs and the solid surface. The chemical reaction caused by CO₂ can, however, be neglected due to the small amount of CO₂ in the chamber.

For Choline amino acid based ILs, the excellent tribological properties are related to the formation of a physically adsorbed film on the metallic surface, which can effectively prevent the friction pairs from direct contact during rubbing. Images of the disc surfaces after friction testing in this work are shown in Figure 4. Based on the stable friction behavior, it is found that the friction can be controlled through moisture when the RH is not higher than 54%. Herein, it is also found that the wear scars in these conditions are barely distinguishable, and the measured surface roughness was approximately the same magnitude as the original lapped surface, i.e., around 50 nm surface roughness ($R_s$). The worn surfaces were investigated by scanning electron microscopy (SEM) as well, shown in Figure S2, Supporting Information. Figure S3, Supporting Information, shows X-ray photoelectron spectroscopy (XPS) spectra gathered from a steel disc surface lubricated with [Cho][Pro] at 25 °C and 54% RH. There are no distinct differences in the chemical characteristics of the surface inside and outside the wear scar. Therefore, no attempt was made to quantify the wear coefficient. At 75% RH, the wear scar was slightly more obvious than those at lower RH. Furthermore, as shown in Figure 4e, the wear became severe at 94% RH, while stable friction could not be reached under this condition. In other words, the failure of friction control under extremely high RH may be caused by the changed interfacial structure.

From the severe wear observed at higher RH, it can be inferred that the tribofilm was broken down during the friction test. The loss of excellent antiwear ability could be caused by the presence of free water at 94% RH, while at lower RH the water state keeps water bound. The presence of water in the ILs also largely affects the interionic interactions and alters their physical properties such as viscosity. After friction test, it was also found that the viscosities of lubricants varied significantly. The viscosities of [Cho][Pro] with different water contents were investigated and Figure 5 shows how the viscosity decreases as a function of water content. The viscosity is relatively insensitive to the shear rate in the measured range when the water content is below 20%.

According to Hamrock–Dowson theory for elastohydrodynamic lubrication (EHL) of point contacts, the minimum film thickness, $H_c$ can be calculated from Equation (1).

$$H_c = 3.63R_e U^{0.68} G^{0.49} W^{-0.073} (1 - e^{-0.68k})$$

Figure 3. Online friction control behavior through humidity: a) absorbing moisture under higher environmental humidity (54% RH); b) evaporating water under lower environmental humidity (20% RH).
where $k$ is the ellipticity parameter and

\[ U = \frac{\eta_0 U_e}{R_x E'} \]  \hspace{1cm} (2)

\[ W = \frac{F_N}{R_x^2 E} \]  \hspace{1cm} (3)

\[ G = \alpha E' \]  \hspace{1cm} (4)

where $U$ is the dimensionless speed parameter, $G$ is the materials parameter, $W$ is the load parameter, $R_x$ is the radius of curvature of the ball in $x$-direction, $\eta_0$ is the viscosity at atmospheric pressure (Pa), $U_e$ is the entrainment speed (mm s$^{-1}$), $E'$ is the equivalent Young modulus (Pa), $F_N$ is the normal force (N), and $\alpha$ is the pressure–viscosity coefficient (Pa$^{-1}$). Except $\alpha$, all of these are known. An approximation of this parameter for [Cho][Pro] at 25 °C is 8.0 GPa$^{-1}$, which was computed in the previous work when the water content was lower than 15%.$^{[40]}$ Although we could not get the exact value of pressure–viscosity coefficient with 20%, 25%, and 30% water content, we know that the pressure–viscosity coefficient will decrease after adding more water into [Cho][Pro].

In reciprocating motion, taking into consideration the changing speed, the minimum film thickness can achieve the largest value when calculated by the maximum sliding velocity. The maximum sliding speed can be calculated from Equation (5)

\[ U_{\text{max}} = \pi f L \]  \hspace{1cm} (5)

where $f$ is the sliding frequency and $L$ is the stroke length.

The lubrication regime can be distinguished by the method of using the film parameter, i.e., the ratio of the theoretical minimum film thickness, and can be calculated from Equation (6)$^{[44]}

\[ \lambda = \frac{h_c}{\sigma} = \frac{h_c}{\sqrt{\sigma_1^2 + \sigma_2^2}} \]  \hspace{1cm} (6)

where $\sigma$ is the combined surface roughness and $\sigma_1$ and $\sigma_2$ are the surface roughness of the ball and disc, respectively.

The calculated minimum film thicknesses and film parameters are listed in Table 2. All the calculated film parameters are smaller than 1, which means that the tests are performed under boundary lubrication and therefore the viscosity difference between the lubricants will have a small effect on friction. Instead, other factors such as adhesion play a significant role in controlling the tribological performance.$^{[45,46]}$

Figure 4. 3D images of worn steel discs at various RHs: a) 8% (KOH), b) 33% (MgCl$_2$), c) 54% (Na$_2$Cr$_2$O$_7$), d) 75% (NaCl), and e) 94% (KNO$_3$).

Figure 5. Viscosity measurements of [Cho][Pro] as prepared and [Cho][Pro] with different water contents.
coworkers have confirmed that the lubricating mechanism of ILs under low load is mainly attributed to the adsorption ability of ions on the substrate surface. Thus, the friction behavior altered by the environmental humidity is probably influenced by the effect of adhesion on the water uptake from humid air by the [Cho][Pro]. The tribological behaviors lubricated by ILs after absorbing small amounts of water have been investigated by several researchers. Although the tribological performance in these studies varied depending on the particular substrate/environment combination and experimental conditions, it has been found that the presence of water in ILs at the interface changes the adhesion, which contributes to altered friction.

Another possible explanation is that the absorbed water in ILs can have a strong impact on the properties of a layer of IL confined between polar counterparts, based on investigation using atomic force microscopy and surface forces apparatus. In particular, in the case of hydrophilic ILs, the large amounts of absorbed water change their organization. Spencer and coworkers found that ambient RH not only leads to significant water uptake by hygroscopic ILs, but also is expected to affect the organization of IL film layers. The layers thickness is increased due to the fact that water hydrates the anion and expands the structure across the gap, characterized by extended surface forces apparatus (eSFA) and colloidal-probe lateral force microscopy (CPM). Later, Espinosa-Marzal and coworkers also found a mild swelling of the nanostructure using wide-angle X-ray scattering, when 1-hexyl-3-methylimidazolium ethylsulfate was exposed to ambient humidity. It could be related to the induced weakening of the electrostatic interactions and hydrogen bonding between the anion and cation by the water molecules. Presumably, this phenomenon could occur also for [Cho][Pro] and polar surfaces, including steel surfaces.

Furthermore, it is also necessary to point out the influence of water concentration on the organization of the IL layers, which is modulated by environmental humidity. As discussed previously, the controllable friction behavior enabled in a wide range of humidity is attributed to the plentiful hydrogen bond donors and acceptors found in [Cho][Pro]. More importantly, Gontrani and coworkers found that the types of hydrogen bonds in Choline-based IL/water mixtures could be determined by the water content, based on their molecular simulation and experimental studies. As schematically shown in Figure 6b, the water molecules prefer to establish H-bonds with the cations and anions in the case of lower water content. With the water concentration increase, IL pair formation decreases and H-bonds between water and water form, as shown in Figure 6c. Thus, the active controllable friction could be attributed to the reversible changes of H-bond types. However, in the case of hydrophilic ILs, the shift of the slippage plane, i.e., from ionic plane to the solid/liquid interface, is still uncertain and needs further investigation.

3. Conclusions
In summary, [Cho][Pro], a green ionic liquid with controllable friction properties has been investigated. The friction control is obtained by altering the environmental humidity. The friction increases at higher RH and it turns lower when partial water is evaporated under lower environmental humidity. As the experiments are performed in boundary lubrication, the modulation of friction is hypothesized to be based on the changing adhesion depending on water content. The analysis of H-bond types modified by the water content could probably provide another explanation of controllable friction.

4. Experimental Section
Materials and Preparation of ILs: In this work, ILs, namely, [Cho][Pro], was synthesized from choline hydroxide solution (46 wt% in water, Sigma-Aldrich, USA) and L-proline (purity > 99.99 wt%) by a method described in a previous study. First, 12.21 g (0.105 mol) proline was added into 26.344 g choline hydroxide solution with 0.1 mol choline hydroxide. The mixture was stirred for 48 h at room temperature. Then, it was washed with ethyl acetate to remove the excessive amino acids. In the end, the obtained mixture was subjected to rotary evaporation under

![Figure 6](www.aem-journal.com)
70°C for 1 h and then dried in a vacuum oven at 70°C for 2 days to get the final products.

Humidity Control: A saturated solution of salt in water can be used to maintain particular values of RH inside containers.[34] In this study, five saturated salt solutions were chosen to control the environmental humidity, listed in Table 1. All of them were purchased from Sigma-Aldrich.

To achieve the targeted RH, the chosen saturated salt solutions were placed in a closed chamber and the humidity inside the chamber was continuously monitored with a commercial humidity sensor (Amprobe TH-1). A schematic diagram of the humidity-controlled tribometer system is given in Figure S1, Supporting Information.

Friction and Wear Tests: The lubricity of the [Cho][Pro] under different humidities was investigated using an Optimol SRV-III oscillating friction test rig. The upper steel ball (52100 bearing steel, diameter 10 mm, surface roughness (R_a) 20 nm, provided by SKF, Sweden) slides under reciprocating motion against a circular disc. The tested disc was made of steel (100Cr6 ESU hardened, Ø24 mm, 7.9 mm, and R_a 120 nm), supplied by Optimol Instruments Prüechnik GmbH, Germany. Before each test, the device and specimens were cleaned with acetone and ethanol. Before each test, 0.03 g [Cho][Pro] was dropped on the disc as lubricant. The tests were conducted under a load of 5 N (0.8 GPa maximum Hertzian pressure) at a sliding frequency of 50 Hz, and an amplitude of 1 mm. The friction coefficient curves were recorded with a data acquiring system linked to the SRV-III tester. After the tests, 3D topography of the dis surfaces and ball surfaces were determined using an optical system (Zygo 7300). It was recorded from the optical interferometric profilometer using 2.75 × optical magnification. SEM images were recorded on an SEM instrument (HITACHI, S-4800). The chemical composition of the wear scars was confirmed by XPS, and the results were acquired on a Thermo K-Alpha XPS, Thermo Fisher Scientific. Before SEM and XPS analysis, the lower discs were cleaned by ultrasonication in acetone for 20 min to remove the residual lubricants on the surface.

Water Content Analysis: The absorbed water amount was obtained by measuring the mass gain with an analytical balance with an accuracy of 0.00001 g.

Viscosity: The viscosities of [Cho][Pro] containing different water contents were measured at 25°C using a CVO Bohlin rheometer. The viscosity samples were measured with the cone/plate geometry. The shear rate was increased logarithmically from 0 to 100 s⁻¹.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

Acknowledgements
The authors thank the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning (Formas, Grant No. 2016-01098, 2019-00904), the Swedish Research Council (Project No. 2017-04914, 2019-04941), the Swedish Energy Agency (Energimyndigheten, Grant No. 2018-003910), and the Mistra Innovation foundation of Sweden (Grant No. MI16.23) for financial support.

Conflict of Interest
The authors declare no conflict of interest.

Keywords
friction control, humidity, ionic liquids, viscosity

Received: October 16, 2019
Revised: December 25, 2019
Published online: February 9, 2020

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