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Effects of humidity on the mechanical properties of gecko setae

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ABSTRACT

We tested the hypothesis that an increase in relative humidity (RH) causes changes in the mechanical properties of the keratin of adhesive gecko foot hairs (setae). We measured the effect of RH on the tensile deformation properties, fracture, and dynamic mechanical response of single isolated tokay gecko setae and strips of the smooth lamellar epidermal layer. The mechanical properties of gecko setae were strongly affected by RH. The complex elastic modulus (measured at 5 Hz) of a single seta at 80% RH was 1.2 GPa, only 39% of the value when dry. An increase in RH reduced the stiffness and increased the strain to failure. The loss tangent increased significantly with humidity, suggesting that water absorption produces a transition to a more viscous type of deformation. The influence of RH on the properties of the smooth epidermal layer was comparable with that of isolated seta, with the exception of stress at rupture. These values were two to four times greater for the setae than for the smooth layer. The changes in mechanical properties of setal keratin were consistent with previously reported increases in contact forces, supporting the hypothesis that an increase in RH softens setal keratin, which increases adhesion and friction.

1. Introduction

Geckos have an amazing ability to run up/down walls or across ceilings with ease using their remarkable toes. Gecko toes are not sticky in the same manner as traditional pressure-sensitive adhesives (PSAs), such as adhesive tape. Instead, the toes are covered with a "smart" adhesive of micro- and nano-structures of β-keratin that adhere strongly yet are non-fouling, non-tacky and detach easily [1,2]. Each toe bears a hierarchical structure composed of a series of ridges bearing hair-like shafts (setae) which divide into hundreds of flat tips called spatula [2], shown in Fig. 1.

Scientific debate over the microscopic mechanism of gecko adhesion has persisted for over two centuries. Unsupported mechanisms include suction, electrostatic attraction, micro-interlocking, and sticky secretions. Capillary adhesion and intermolecular van der Waals (vdW) attraction remain possible mechanisms [1,2], although we discovered that van der Waals force is sufficient to explain gecko adhesion [3]. Since vdW force is largely independent of surface chemistry, this discovery paved the way for the development of synthetic gecko adhesives from a variety of materials [4,5].

A vdW mechanism does not rule out a role for water in gecko adhesion, and other studies have shown that adhesion increases at high relative humidity (RH), raising the possibility that surface hydration effects may contribute to adhesion [6–8]. These effects are presumed to be strictly capillary [8] or the result of surface energy modification [6]. In contrast, we recently demonstrated that capillary forces cannot explain the effect of RH on gecko adhesion and proposed that changes in mechanical properties explain the increase in adhesion at high humidities [9], a prediction also made by Chen and Gao [10]. This paper presents further measurements of the effects of humidity on the mechanical properties of gecko setae and β-keratin.

The mechanical properties of mammalian α-keratins are strongly influenced by moisture [11–16]. The avian and reptilian form, β-keratin, has been studied to a lesser extent [17–19], with similar results. There has been only limited mechanical testing on tokay gecko setae [20,21]. Peattie [21] performed a vibrational analysis to determine the elastic modulus (E) of a single seta to be 1.4–1.6 GPa. Huber et al. [20] used nano-indentation (E = 1.2 GPa), tensile testing (E = 7.3 GPa) and three-point bending (E' = 1.7 GPa). Huber et al. [21] did not observe an effect of humidity on the setal modulus, but since their samples were prepared under high vacuum, permanent dehydration of the keratin cannot be ruled out as an explanation for the absence of subsequent humidity effects [22].

The work of adhesion is approximately 50 mJ m^{-2} for vdW interactions [23], but as a result of viscoelastic energy loss the work of detachment of PSAs can be orders of magnitude greater [24,25]. The elastic modulus of the material also greatly affects adhesion, according to guidelines presented by Dahlquist [26], as

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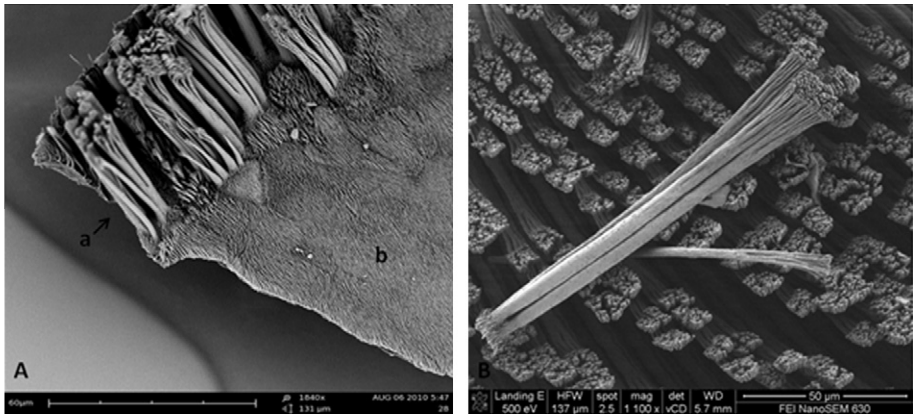

Fig. 1. (A) SEM image of (a) a setal array and (b) the smooth epidermal lamellar layer. (B) SEM image of isolated tokay gecko setae. The setae are approximately 120 μm in length, with an average diameter of 3.3 ± 0.2 μm. The setae branch forming numerous flattened spatula tips.

the upper limit of Dahlquist's criterion for tacky materials is 100 kPa at 60 Hz. Interestingly, the effective modulus of setal arrays is 80–100 kPa [27]. This suggests that any changes in the elastic modulus of the bulk setal keratin will be inversely related to adhesion: setae with lower elastic moduli should be stickier, and stiff setae may not adhere at all. While a low E is beneficial for adhesion, it may promote matting of the setae [29].

It is well established that the complex modulus decreases with an increase in water content, along with an increase in loss tangent (viscoelastic loss) for some structural proteins [22], including α-keratin [14–16]. However, no data exist for gecko β-keratin. In this study we have measured the effects of humidity on the dynamic mechanical response and tensile properties of isolated gecko setae. Because preparation and testing of a single gecko seta is time consuming, we also tested 0.75×1.1 mm strips of smooth epidermal layer cut from isolated seta-bearing gecko lamellae to determine whether a macroscopic lamella can be used as a proxy for microscopic setae in future mechanical testing.

2. Materials and methods

We collected setal arrays from the hind center toe of an adult male and female tokay gecko (*Gekko gecko*) and separated single seta from the lamellar backing. We cut strips of the smooth, outer epidermal layer of approx. 0.75×1.1 mm from setal arrays collected in the same fashion as above. SEM was used to determine the setal diameters (3.3 ± 0.2 μm) while a high magnification CCD camera system was utilized to determine the tested setal length and lamella dimensions.

We used a specially designed mechanical testing apparatus at Lewis and Clark College [28]. The apparatus is housed in an environmental chamber (Electro-Tech Systems Inc. model 5503-00) with controllable temperature and humidity (0–80% RH). A single axis quartz piezoelectric load cell is mounted opposing a two-axis Aerotech (Pittsburgh, PA) model ANT-50L motorized stage. A type 9207 load cell (Kistler, Winterthur, Switzerland) and an analog to digital amplifier capable of 0.084 mN resolution was used for the individual seta testing. A Kistler three-axis piezoelectric load cell with resolution of 1.3 mN was used to test the lamella. The system is capable of the fine movement and load measurements needed to measure the tensile forces of single seta.

The gecko setae were fixed to the end of a fine 0.1 mm diameter stainless steel minutien pin with cyanoacrylate glue. The pin was then glued to a glass microscope slide, rigidly mounted on the two-axis stage. A small amount of glue was used to secure the free end of the seta to the load cell. A similar method was employed to secure the lamella, except the lamella was secured directly to the glass slide.

In dynamic mechanical analysis (DMA) a specimen is subjected to sinusoidal strain with amplitude ε_0 and frequency ω: $\varepsilon(t) = \varepsilon_0 \sin(\omega t)$. The resulting dynamic stress, $\sigma(t) = \sigma_0 \sin(\omega t + \delta)$ will be phase shifted with respect to the strain by the loss angle δ. The dynamic material behavior can be described by the magnitude of the complex modulus E^*:

$$|E^*| = \sqrt{(E')^2 + (E'')^2} = \frac{\sigma_0}{\varepsilon_0} \qquad (1)$$

where E' and E'' are the storage modulus and loss modulus, respectively, and are related by the dimensionless loss tangent:

$$\tan\delta = \frac{E''}{E'} \qquad (2)$$

We conducted our tests at three frequencies (ω = 0.5, 5, and 10 Hz), over a range of humidities (RH = 10%, 20%, 30%, 40%, 50%, 60%, 70%, and 80%), and, due to limitation of the Aerotech stage for sinusoidal movement of 10 Hz at amplitudes of <1.5 nm (strain <2%) required for setae testing, a nominal ε_0 = 2% was used for all setae DMA testing. For consistency, a nominal ε_0 = 2% was also used for the lamellae DMA testing. Tensile testing of individual seta and lamella was also carried out using the same experimental setup as the DMA testing, but were strained to failure at RH of 30% and 80%. The strain rate under tensile strain was 2% s^{-1}.

3. Results and discussion

Fig. 2 shows the stress–strain data obtained from individual seta. The force of deformation was linear for <1% strain. The elastic modulus was 3.7 ± 0.1 GPa at 30% RH, with rupture at ε_f = 6.8 ± 0.2% and σ_f = 262.5 ± 3.5 MPa. In setae at 80% RH E = 2.13 ± 0.2 GPa, with rupture at ε_f = 16.7 ± 1.0% and σ_f = 237 ± 22 MPa.

We measured single seta under dynamic sinusoidal loading (Fig. 3) and determined the magnitude of the complex modulus ($|E^*|$) at 80% RH to be ~1/3 the value in dry conditions, a decrease in E^* from 3.1 ± 0.1 to 1.23 ± 0.15 GPa. Additionally, the loss tangent increased by over 3-fold with humidity, from $0.055 \pm 8 \times 10^{-3}$ at 30% RH to 0.173 ± 0.015 at 80% RH, indicating an increase in viscoelastic loss with water absorption. There was no statistical difference observed for both E^* and tan (δ) over the frequency range tested (ω = 0.5, 5, and 10 Hz).

Stress at rupture for the lamella was significantly less than for the seta, suggesting that lamellae cannot be substituted for setae in all circumstances. This distinction was most pronounced at 80% RH. The cause of the difference is unknown, but we surmise