Review

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Authors for correspondence:

Longjian Xue

e-mail: xuelongjian@whu.edu.cn

W. Jon. P. Barnes

e-mail: jon.barnes@glasgow.ac.uk

Tree frog adhesion biomimetics: opportunities for the development of new, smart adhesives that adhere under wet conditions

Fandong Meng¹, Quan Liu¹, Xin Wang¹, Di Tan¹, Longjian Xue¹ and W. Jon. P. Barnes²

¹School of Power and Mechanical Engineering, Wuhan University, South Donghu Road 8, Wuhan, People's Republic of China

²Centre for Cell Engineering, University of Glasgow, Joseph Black Building, Glasgow G12 8QQ, UK

WJPB, 0000-0002-1359-5803

Enlarged adhesive toe pads on the tip of each digit allow tree frogs to climb smooth vertical and overhanging surfaces, and are effective in generating reversible adhesion under both dry and wet conditions. In this review, we discuss the complexities of the structure of tree frog toe pads in relation to their function and review their biomimetic potential. Of particular importance are the (largely) hexagonal epithelial cells surrounded by deep channels that cover the surface of each toe pad and the array of nanopillars on their surface. Fluid secreted by the pads covers the surface of each pad, so the pads adhere by wet adhesion, involving both capillarity and viscosity-dependent forces. The fabrication and testing of toe pad mimics are challenging, but valuable both for testing hypotheses concerning tree frog toe pad function and for developing toe pad mimics. Initial mimics involved the fabrication of hexagonal pillars mimicking the toe pad epithelial structure. More recent ones additionally replicate the nanostructures on their surface. Finally we describe some of the biomimetic applications that have been developed from toe pad mimics, which include both bioinspired adhesives and friction-generating devices.

This article is part of the theme issue 'Bioinspired materials and surfaces for green science and technology (part 2)'.

1. Introduction

Evolution through natural selection has, over many millions of years, produced structures in animals and plants that are superbly adapted to their functions. It is therefore of no surprise that scientists have looked at the natural world for inspiration in solving complex human problems, a field known as bioinspiration or biomimetics [1]. Thinking particularly of surfaces, we have, for example, developed swimsuits with increased drag reduction to enhance swimming efficiency, based on the pattern of dermal denticles (skin scales) that cover the skin of fast-swimming sharks [2] and Lotusan, a self-cleaning exterior paint, based on the superhydrophobic surface of lotus leaves [3]. Many more examples are described in a recent review by Sun & Bhushan [4].

The adhesive mechanisms of climbing animals have clear-cut implications for biomimetics. For example, they adhere well to many surfaces, adhesion is reversible so that the adhesive is re-usable, and only stick when required [5,6]. Additionally, they self-clean, so that they are resistant to failure through the accumulation of dirt particles [7–9]. Initially, work was mainly carried out on geckos [5,10,11], because they are among the heaviest animals using reversible adhesion for their locomotion and have an amazing ability to run across ceilings. These studies have shown that adhesion is mainly (if not entirely) due to van der Waals forces, as the extremely small tips (spatulae *ca.* 200 nm wide and 5–20 nm thick) of the highly branched adhesive setae (hairs) on the toe pads of geckos are able to achieve extremely close contact with the surface to which the gecko is adhering. A number of gecko-inspired adhesive structures have been designed and some can support the weight of a human [12]. However, such gecko-inspired structures are difficult to mimic and their durability is far from satisfactory.

The adhesion mechanism of tree and torrent frogs is quite different, which means the biomimetic applications arising from it will differ from those developed from geckos. As will be described in this review, tree frogs mainly adhere by wet adhesion, as there is a thin layer of fluid between the toe pad and the adhering substrate. The adhesive forces are thought to be capillarity and viscosity-dependent hydrodynamic forces [13–15], but a role for van der Waals forces cannot be excluded [16]. Tree frog toe pads also generate friction forces; indeed, maximum friction forces may exceed those arising from adhesion [17,18]. As we shall describe, the tree frog's adhesive mechanism provides good adhesion and friction on soft wet surfaces, and, as a result, is likely to have many important applications, particularly in the field of medicine.

A significant body of work has also been carried out on insects. Insects may have hairy pads like geckos (e.g. beetles and flies) or smooth pads like tree frogs (e.g. cockroaches and grasshoppers) [19]. As there is fluid between pad and external surface, it has, until recently, been assumed that insects, like tree frogs, adhere by wet adhesion. However, Labonte & Federle [20] have recently questioned this conclusion as their most recent data could be better explained by adhesion using van der Waals forces.

In this short review, we will discuss the complexities of the structure of tree frog toe pads in relation to their function as well as the development of artificial toe pad analogues. As we will make clear, studies of the properties of such artificial structures provide insights into the functioning of actual toe pads as well as assisting the development of new smart adhesives based on tree frog adhesive and friction mechanisms. Additionally, we will describe some recent applications of the tree frog's mechanisms of adhesion and friction and discuss the current challenges and future perspectives of this exciting field of research.

2. Tree/torrent frog adhesion

(a) Toe-pad structure and function

Adhesive toe pads are thought to have evolved separately by convergent evolution in several different families of frogs [21]. Most of these families are arboreal, living in trees or shrubs (tree frogs such as *Hypsiboas boans* (figure 1)). However, adhesive pads are also found in frogs living in the region of waterfalls (torrent frogs of the family Ranidae). The adhesive pads are located on

Figure 1. *Hypsiboas boans* is the largest tree frog found in Trinidad (West Indies), with a snout/vent length of *ca* 100 mm (a). Living high in trees, it is a gliding frog, as can be seen by the presence of webbing between the digits of the fore-limbs. This frog has just landed on a bamboo stem, and has slipped downwards, leaving behind some of the watery fluid that forms its adhesive joint (b) (Allan L, 2013, personal communication).

the ventral surface of the toes (toe pads), with related structures (sub-articular tubercles) being located on the ventral surface of more proximal digits. The latter is mainly used in attachment to small diameter structures such as twigs [22]. Features important to the functioning of the toe pads are highlighted below:

Grooves surround each toe pad (circumferal and proximal grooves). These grooves (shown in figure 2b) will divert water around the pad in the wet environments (e.g. rain forests) in which the majority of tree frog species live. Such structures are particularly important in torrent frogs, which would otherwise be washed downstream by the water that covers the rocks on which they are found [24].

The toe pads are epithelial structures, consisting of several layers of cells [14,25,26]. Their development can be clearly seen by the examination of transverse sections of the toes, where the deepest layers are the least specialized and the outermost the fully developed pad cells. At intervals, the outermost layer is shed, the layer beneath becoming the new surface layer (figure 3). In this way, the frog can maintain fully functional toe pads throughout its adult life.

Toe pads are extremely soft structures, aiding close contact to external surfaces and enhancing both adhesion and friction. Indentation experiments using spherical indenters of 264 and 1500 μm diameter [27] show a gradient of elastic modulus from 4 to 25 kPa, the higher values being found in the outermost, keratinized, layers. Such low values for the elastic modulus are comparable to sea anemone mesoglea or jellyfish jelly ($E \approx 3$ kPa [28]). The elastic modulus of a surface can also be calculated from force/distance curves produced by an AFM indenter [29]. Using this method, estimates of the elastic modulus of the external surface of the pad (the method does not provide information on deeper structures), are significantly higher, giving a median value of 5.7 MPa. Surface structures will thus have an increased resistance to wear. By contrast, the innermost layers, lying close to sub-dermal lymph spaces and a capillary network, have much lower elastic moduli and thus are extremely soft and pliable. Keratin filaments run inwards from the surface (figure 3) into the pad allowing the pad to maintain its shape in spite of the softness of the pad material [18,24,30,31].

Figure 2. *Litoria caerulea* toe pads. (a) frog; (b) toe pad surrounded on three sides by a groove (black line); (c) polygonal (mostly hexagonal) epithelial cells surrounded by deep channels; (d,e) the nanopillars that cover the surface of the epithelial cells, shown in surface view (SEM) and section (TEM). Reproduced from Federle *et al.* [23].

Figure 3. TEM images of toe pad epithelium of *Stauroids parvus*. (a) Outer cell layer showing nanopillars and dense bundles of keratin filaments (arrows). (b) Border between outer cell layer on left and the second layer of cells which also have keratin filaments; arrows show invaginations of the cell membrane, which will become the gaps between the nanopillars by the time this layer becomes the outermost layer. Reproduced from Drotleff *et al.* [24].

The actual epithelial cells (the outermost layer), have a complex structure. As described below, they are mainly hexagonal in shape and are surrounded by deep, fluid-filled channels (figure 2c). These channels are thought to serve two functions. First, they help spread the fluid over the entire pad surface, so that there are no air pockets that would reduce adhesion. Second, under wet conditions, they would help to get rid of excess fluid which would reduce adhesion by increasing the separation of pad and surface. The epithelial surface is not flat, but consists of a dense array of nanopillars, 300–500 nm in diameter and 200–300 nm in height (figure 2d,e) [29]. As figure 4

Figure 4. Cryo-SEM images of nanopillars of *Stauroides parvus* (a,b) and *Rhacophorus prominans* (c). The nanopillars are filled with keratin fibrils (arrows in b) and have a concave top (c). Image (a) shows the tops of the nanopillars standing clear of the surrounding frozen fluid (ice). The arrows in (c) show ice partially filling gaps between the nanopillars. (a,b) reproduced from Drotleff *et al.* [24]; (c) is reproduced from Scholz *et al.* [29].

shows, the nanopillars are filled with keratin filaments (figure 4b) and have a concave upper surface. This opens the question as to whether they could act as miniature suction discs, but this has yet to be investigated. There are thus two sets of structures (the cells separated by grooves and the nanopillars) that allow the pad to keep close contact to rough as well as smooth surfaces, promoting both adhesion and friction forces.

The watery pad fluid has a viscosity of $1.25\text{--}1.51\text{ mPa s}^{-1}$. Using a laser tweezer technique, Federle *et al.* [23] have shown that the fluid secreted by the toe pads has a low viscosity in the range $1.25\text{--}1.51\text{ mPa s}^{-1}$ (figure 5). It is usually referred to as mucus, but detailed chemistry remains to be identified. It is secreted by glands within the toes and passes to the pad surface through narrow ducts which open into the grooves that separate the pad epithelial cells. It completely fills the space below the pad and forms a meniscus around the edge of the pad, producing capillary forces [14,32]. The fluid is, however, known to contain molecules that act as surfactants [33], lowering surface tension and thus allowing capillary adhesion to hydrophobic as well as hydrophilic surfaces (e.g. surfaces of waxy leaves).

The fluid layer below each epithelial cell has a thickness of the order of a few nanometres. Federle *et al.* [23], using interference reflection microscopy, have shown that the fluid under the toe pads has a low thickness, except around the edges of the epithelial cells, where the channels between the epithelial cells are located (figure 6). This means that, in addition to capillary forces, substantial velocity-dependent hydrodynamic forces will be present [34]. Indeed, torrent frogs can adhere in running water when the pads are completely submerged, a situation where capillary forces would be absent [35,36].

Tree frog toe pads produce higher friction than adhesive forces, a surprising finding for a fluid joint. Federle *et al.* [23] have shown that it is due to the nanopillars, which form extremely close contact with the external surface (in the range $0\text{--}5\text{ nm}$ (figure 6d)). Nanopillars cover the surface of all the pad epithelial cells (figures 2 and 4), but are absent from other, non-adhesive areas of the skin

Figure 5. Viscosity measurements of *Litoria caerulea* toe pad mucus using laser tweezers. (a) Bead displacement elicited by sinusoidal fluid movement at three different frequencies. (b) Relationship of bead displacement amplitude and velocity (frequency) measured for toe pad mucus and pure water. The difference in the slopes indicates the viscosity. Reproduced from Federle *et al.* [23].

surface. However, it is important to note that the presence of these areas of very close contact means that molecular mechanisms of adhesion cannot be excluded [16].

Lymph spaces whose profiles are easily deformed by the slightest application of pressure. These spaces lie in the dermis below the toe pads and contribute to their remarkable softness and high deformability. Under pressure, they increase the contact area and improve conformation to the underlying surface topography [31], thus promoting both adhesion and friction.

Pad detachment occurs by peeling from the proximal edge of the pads during both forward walking and climbing [37], a mechanism requiring rather small forces (less than 10 mN).

Self-cleaning. Force measurements on both unrestrained free-walking frogs and individual toe pads [8] show that, following contamination by glass beads, toe pad adhesive forces recover after a few steps. Both shear movements and a flushing effect of the secreted mucus play important roles in shedding contaminating particles.

Adhesion to rough surfaces. In their natural habitat, tree frogs need to be able to adhere to surfaces of various roughnesses, including leaves, rocks and bark. Tests in a laboratory setting demonstrate that both adhesion and friction are maintained [38] or increased [39] on micro-rough surfaces (asperity size 0.1–30 μm) and only show a significant decline when asperity size reaches 50 μm. Crawford *et al.* provide evidence that, on these roughest surfaces, the pads secrete insufficient fluid to fill the space under the pad, leaving air pockets that would significantly reduce the

Figure 6. Interference reflection microscopy (IRM) used to measure the thickness of the fluid layer under a toe pad of a living, adhering tree frog (*Litoria caerulea*). What one sees is the interference pattern resulting from the interference of light reflected from the surface of the pad with light reflected from the top surface of the glass to which the frog is adhering. By comparing images of the same pad with green and blue monochromatic light, one can identify which fringe is which, and thus estimate the thickness of the fluid layer under each pad epithelial cell. (a) A representative IRM image and (b) the calculated thickness of the fluid layer along the white arrow in (a). Note that this arrow goes through the middle of an epithelial cell, and that the thickness of the fluid layer at these points is very small. (c) Opening the aperture of the microscope reduces the depth of focus so that only zero-order fringes are seen, allowing a more accurate measurement of pad/ground distances under the pad epithelial cells. As shown in (d), 41% of the epithelial cells have an average fluid film thickness in the range 0–5 nm. Reproduced from Federle *et al.* [23].

Laplace pressure component of capillarity [39]. However, the conflicting results on the lower roughnesses leave unanswered the question of whether the low elastic modulus of the toe pads allows them to mould themselves to the contours of all but the largest asperities. However, as Crawford *et al.*, using interference reflection microscopy, were able to observe that small glass beads (less than 5 μm diameter) could become lodged in the channels between the cells, it is clear that the nature of the epithelium (cells surrounded by channels) allows interdigitation of asperities on the external surface and the toe pad epithelium.

Similarity of toe pad structure in different families of tree frogs. There are extraordinary similarities between the structure of toe pads in different frog families (hylid and rhacophorid frogs have been compared in detail by Barnes *et al.* [30]). They have important implications for biomimetics, for such convergent evolution suggests a good starting point for attempts to develop adhesives that will function in wet conditions.

(b) Mechanisms of adhesion and friction and the forces they produce: physical principles

Although the dominant force in tree frog adhesion is thought to be capillarity, viscosity-dependent hydrodynamic forces are also considered to play a role. Additionally, since the

Figure 7. Capillarity model of sphere on a plane surface connected by a drop of fluid. Redrawn from Endlein & Barnes [6]. (Online version in colour.)

thickness of the fluid layer under each toe pad epithelial cell is less than 35 nm [23], it is likely that there is actual contact between nanopillars on the pad surface and asperities on the substrate. Thus, van der Waals forces will also be involved, but to what extent is unknown. The physical principles underlying capillarity and hydrodynamic forces are outlined below.

Wet adhesion—capillary forces. A common model that has been used for quantifying capillary forces consists of a sphere on a plane surface, connected by a drop of liquid (figure 7). In this model, appropriate as toe pads are slightly domed, the surface tension of the meniscus results in a pressure difference, where the pressure inside the liquid is lower than it is outside, so long as the meniscus is concave. This pressure difference, the Laplace pressure, will resist the separation of the two surfaces. The attractive Laplace force for a macroscopic, perfectly smooth and homogeneous sphere in contact with a plane is given by

$$F_L = -2\pi R\gamma(\cos \theta_1 + \cos \theta_2), \quad (2.1)$$

where R is the radius of the sphere, γ is the surface tension of the liquid, and θ_1 and θ_2 are the contact angles of the liquid with the plane surface and sphere, respectively. If the liquid completely wets both surfaces (i.e. θ_1 and θ_2 both equal 0), equation (2.1) simplifies to

$$F_L = -4\pi R\gamma. \quad (2.2)$$

An alternative model examines capillary forces between two rigid plates, separated by a thin layer of fluid (figure 8). This model produces substantial forces if the radius (r) of the area of contact is large and the separation distance of the plates (h) is small, according to the following equation:

$$F_L = \pi r^2 \gamma [r^{-1} - (\cos \theta_1 + \cos \theta_2)h^{-1}]. \quad (2.3)$$

In a large flat meniscus where $r \gg h$ and $\theta_1 = \theta_2 = 0$, equation (2.3) approximates to equation (2.4), namely

$$F_L \approx -\frac{2\pi r^2 \gamma}{h}. \quad (2.4)$$

A second component of the adhesive force is the tensile force of the meniscus (figure 9). In the rigid plate model, this surface tension force is

$$F_T = -2\pi r\gamma. \quad (2.5)$$

Figure 8. Capillarity model of two flat, rigid surfaces separated by a small volume of fluid. Redrawn from Endlein & Barnes [6]. (Online version in colour.)

Figure 9. Viscous force model of two flat rigid plates, fully immersed in a fluid, subject to an external force. Redrawn from Endlein & Barnes [6]. (Online version in colour.)

The total meniscal force (F_M) is the sum of F_L and F_T

$$F_M = F_L + F_T, \quad (2.6)$$

where F_T is negligible in any single large meniscus (e.g. ones with a radius of 0.5–3 mm as occur in tree frog toe pads), but would dominate adhesive forces in a fly's adhesive pad where you have large numbers of microscopic menisci (radii of *ca* 1 μm).

In a recent study [40], equation (2.4) has been extended from hard, undeformable surfaces and spheres to soft, elastic materials, such as the toe pad of a tree frog. The mathematical equations relate the capillary attraction between the bodies to their elastic repulsion. Although they do not lend themselves to any easy calculation of the adhesive force, they are of interest in that they predict that F_M scaling will gradually change from length scaling to area scaling with increasing r , this change occurring more rapidly for materials with a lower effective elastic modulus (E_{eff}). For instance, in equation (2.7) which deals with the Laplace pressure component of adhesion, the first term is proportional to r and is identical to the force with a hard sphere (equation (2.4)), while the second term is proportional to r^2 . It is negligible for high Young's moduli, but becomes significant for soft materials.

$$F_L = -4\pi r\gamma - \left(\frac{\pi\gamma}{2r'}\right)^3 \cdot \frac{2r^2}{3(E_{\text{eff}})^2}, \quad (2.7)$$

where r' is the radius of curvature of the meniscus.

Wet adhesion—viscous forces. The second component of wet adhesion is provided by viscosity-dependent hydrodynamic forces, usually referred to as Stefan adhesion. Consider two rigid plates fully submersed in a fluid (figure 9). Separating the plates involves fluid flowing into the gap between them, so that separation is resisted by a viscous force until the fluid movements are complete. This hydrodynamic force (F_V) will be greater for more viscous fluids and for smaller values of h . As equation (2.8) indicates, F_V scales with area squared.

$$F_V = -\frac{dh}{dt} \cdot \frac{3\pi\eta r^4}{2h^3}, \quad (2.8)$$

where η is the viscosity of the liquid and t is the time needed to separate the two plates. For completeness, the equation for the hydrodynamic force between a sphere and a plate (equation (2.9)) is also included.

$$F_V = -6\pi\eta r \frac{dh}{dt} \left(1 + \frac{r}{h}\right), \quad (2.9)$$

when the sphere touches the plate, as in figure 1, $h=0$.

Viscous-poroelastic adhesion. Recently, Tulchinsky & Gat [41] have introduced the new concept of ‘temporary adhesion’ by ignoring inertial and capillary effects, based on the interaction between elastic deformation and viscous flow. During tests on a ‘model toe pad’, the pad generated forces that resisted slip for periods in excess of 200 s. This is fine for a moving frog, but could present problems for a frog at rest. Also, as the fluid used in the model toe pad (silicone oil) had a viscosity several orders of magnitude greater than tree frog mucus, it remains unclear whether the mechanism is applicable to frogs.

Toe pad adhesion and friction forces. Tree frogs are good climbers, many tropical species being found high in the canopy of rain forests. On effectively flat surfaces (e.g. smooth tree trunks), adhesion is the only means by which tree frogs can climb without falling, but on smaller diameter structures (e.g. small stems/twigs), they can additionally climb using adduction forces by grasping around these structures with their digits [22]. Indeed, as Hill *et al.* describe, climbing is rapid on small diameter smooth surfaces because adduction and adhesion act together, and the subarticular tubercles are also brought into play. Unlike geckos, tree frogs cannot run across a ceiling, but they can climb vertical and overhanging surfaces, and a small tree frog can hang on to an inverted glass plate using just its toe pads [32]. At rest, thigh and belly skin aid adhesion.

Quantitative measurements of the adhesive forces that can be generated by tree frogs have been made by a simple procedure first developed by Emerson & Diehl [14]. Frogs are weighed and then placed ‘head-up’ on a smooth surface on a rotation platform that is rotated slowly from 0° (horizontal), through 90° (vertical) to 180° (upside-down). The angles at which the frog falls from the surface (fall angle) are recorded. These fall angles are used to calculate maximum adhesive forces by simple trigonometry [32]. If the total toe pad area is also measured, these values can be converted to the force per unit area that toe pads can generate. Figure 10*a* shows the masses of 14 different frog species (12 of which were hylids). Measured total toe pad areas of these same frogs appear in figure 10*b*, while angles of fall from the rotation platform for these 14 frog species (means of 10 measurements for each frog) are plotted in figure 11*c*. Since frogs have an area-based adhesive system (the toe pads), it is unsurprising that larger (and hence heavier) species fall from the platform at lower angles. From this information, the force per unit area of these toe pads can be calculated (figure 11*d*). The positive slope of the line of best fit, though small, is statistically significant ($r=0.68$; d.f. = 11; $p < 0.05$), and reflects the fact that larger tree frogs have more efficient toe pads. A similar study, but looking at the effects of growth on adhesion of these same species [43], shows similar effects, toe pads becoming more structurally complex as the frogs grow. Close examination of the behaviour of tree frogs during such tilting experiments [42,44] demonstrates that tree frogs have evolved special strategies to maintain adhesion on overhanging surfaces. As the angle of tilt increases, tree frogs spread their limbs out sideways, and to a more limited extent, forwards or backwards as well. As figure 11 shows, this reduces the angle of

Figure 10. Allometric relationships of 13 hyliid species (open circles) and two non-hyliids (filled triangles) plotted against snout–vent length (SVL). (a,b) Morphometric relationships between frog mass, toe pad area and length (log : log plots). (c) Fall angle against length (log : linear plot). (d) Force per unit area plotted against length (log : log plot). Modified from Barnes *et al.* [32].

Figure 11. (a) Video-image of ventral view of a tree frog (*Hypsiboas boans*) clinging to an overhanging translucent surface, with both fore and hind limbs stretched out sideways. Only areas in contact with the surface are in sharp focus. Scale: large squares are 10 mm across. From Barnes *et al.* [42]. (b) Explanatory model.

contact between pad and surface, reducing the tendency to peel as the mass of the frog is now supported by friction as well as adhesive forces.

In recent years, the development of miniature force plates that can measure friction and adhesive forces from single toe pads have allowed many interesting findings. As well as providing direct measurements of the friction and adhesive forces that toe pads can produce (see

Figure 12. Single pad adhesion and friction forces in the tree frog, *Litoria caerulea*, before and after adding a drop of water to the pad. This abolished the meniscus around the edge of the toe pad and resulted in a small increase in pad/ground distance. Hence the reduction in both adhesive and friction forces. Typical pad forces were of the order of 1 mN mm^{-2} . Barnes WJP & Federle W, 2006.

below), experiments can be carried out that test different possible mechanisms by which toe pads can adhere.

Figure 12 shows the results of a simple experiment designed to examine the role/importance of capillarity in tree frog adhesion (Barnes WJP & Federle W, 2006). Both friction and adhesion components of the resultant force are shown for a 70° pull-off, which followed a short initial slide, designed to ensure good initial contact of pad and force plate. A drop of water was then gently added, covering the pad in such a way that the meniscus around the edge of the pad was abolished. As expected, adhesive forces fell to low levels, but recovered following removal of the water (not shown). What was not expected was that friction forces declined as well. The only possible conclusion, supported by subsequent experiments examining the effects of the procedure on the thickness of the fluid layer under the pad using interference reflection microscopy, was that this simple procedure had increased the pad–ground distance. Such a change would of course also have reduced adhesion from viscosity-dependent hydrodynamic forces, and also molecular interactions (e.g. van der Waals forces) that depended on close contact of pad and surface. The experiment is, however, interesting, even though it did not confirm the dominant role of capillarity that it was intended to test. This is because it demonstrates another, equally important role for capillary forces in holding the pad close enough to the ground to allow the other forces to exert their adhesive action.

A second interesting result resulting from single pad force recordings comes from the work of Federle *et al.* [23] in which friction forces were recorded during a short horizontal pull. Since friction forces developed before the pad began to slide and, additionally, there was a ‘remaining’ friction force 2 min after the slide was completed, it is clear that toe pads can generate static friction, strongly suggesting that, during such times, there is actual contact between structures on the toe pads (presumably the nanopylls) and the force plate.

Finally, as has been made clear from the above, these miniature force plates are particularly suitable for measuring the maximum adhesive and friction forces that tree frog toe pads can produce, since both the maximum force and the area of contact are easy to measure. In the gliding frog, *Rhacophorus dennysi*, the calculated values were $1.45 \pm 1.14 \text{ mN mm}^{-2}$ ($N = 33$) for adhesive force and $6.21 \pm 5.11 \text{ mN mm}^{-2}$ ($N = 24$) for friction [17]. This result supports one of the main

conclusions of Langowski *et al.* [16], that toe pads are optimally developed for friction rather than adhesion.

3. Biomimetics of tree frog adhesion and friction

Learning from biology to develop new materials and devices, aiming to solve problems in daily life, medical, industry, etc., comes along with human history, though the word ‘biomimetics’ is a relatively new word, coined by Otto Schmitt in 1957. The mimicking of the clinging abilities of geckos and tree-frogs are always attractive for people, so that animal-inspired adhesives have aroused more and more attention in recent years. However, gecko-inspired materials/devices have caught most of the attention because the origin of gecko adhesion is considered mainly to be due to van der Waals forces so that gecko-mimicking can simply focus on the design and construction of pillar-like structures with various materials and dimensions. Though simple hexagonal structures are present on the toe pads of tree frogs, they are covered by an array of nanopillars and the mechanism of its adhesion is much more complicated, as described above.

Though the toe pads of tree frogs look quite different from the hairy gecko pads (figure 2a), they both can be considered as pillar arrays [45]. A variety of manufacturing technologies [4] have been developed to construct pillar arrays in materials with Young’s moduli ranging from 1TPa to several MPa (for instance, 1TPa of carbon nanotube (CNT) [46], several GPa of polymers like polyurethane (PU) [47], polystyrene [48], polystyrene-block-poly(vinyl-2-pyridine) (PS-b-P2VP) [49] and 1–3 MPa of polydimethylsiloxane (PDMS) [50]). Because of the huge difference in Young’s modulus, pillar arrays with different materials can produce different aspect ratios (AR), which is vital for the stability and adhesion performance of pillar arrays.

(a) Influences of aspect ratio of pillar

Though CNT possesses a large Young’s modulus of 1TPa, the CNT forest shows a much smaller effective modulus (E_{eff}), which allows CNT forests to maximize contacts, increasing the adhesion force [51]. CNT-based adhesives showed shear adhesion of approximately 100 N cm^{-2} , 10 times higher than that of gecko foot-hairs [46]. The complicated fabrication process and the poor durability of CNT forest, however, hinder its future applications. By contrast, polydimethylsiloxane (PDMS), which has a relatively low modulus, has been widely used to construct structured adhesives due to its handling simplicity and commercial availability. Compared to the flat surface, PDMS micropillar arrays showed a remarkable decrease in E_{eff} , so that a larger AR results in a smaller E_{eff} (figure 13a,b). The decreased E_{eff} results in a higher compliance to the contact surface, causing a higher elastic energy dissipation during pull-off from the surface [52]. For instance, increasing the AR of PDMS micropillar arrays from 0.5 to 4 increased pull-off forces by a factor of 3 [53]. It is worth mentioning that measurement of the E_{eff} of a pillar array with a small AR (e.g. 0.5) could be influenced by its backing material. However, higher ARs are not always beneficial for adhesion enhancement. In the case of frog-inspired hexagonal pillars with height of H and side length of L (figure 13c), the adhesion force was found to decrease when H/L was increased from 0.75 to approximately 1.9 (figure 13d) [54]. Similar adhesion dependence on the pillar AR was also reported by Iturri [55]. It was explained that the pillars tend to bend and cluster at a high AR, due to the small gap between pillars. The bending and clustering may contribute to the adhesion reduction in two possible ways: (1) the bending of pillars offers the pillar edge for contact, reducing the effective contact area; (2) the clustering of pillars may form a continuous area for contact, reducing the effect of contact splitting. Moreover, the contact density will be reduced by increasing the channel width W , resulting in a smaller adhesive force. This may explain why tree frog toe pads have a small pillar height and a small channel width.

Though the adhesive toe pads on tree frogs and bush-cricket are normally called smooth adhesive pads, the pillar tops are never smooth. As described above, concave nanostructures have been found on the toe pads of tree frogs (figure 4), and peg-structures have also been reported in bush-cricket [56]. Li *et al.* [57] reported the hexagonal micropillars with

Figure 13. (a) SEM image of pillar arrays. (b) The dependence of effective elastic modulus of PDMS micropillars on the AR. (c) Image of hexagonal pillars in top view. (d) The dependence of the wet adhesion force on the height-to-length H/L . (a,b) reproduced from Greiner *et al.* [52]; (c,d) are reproduced from Wang *et al.* [53].

micro-bulges on pillar top prepared by combining hemispheric crater arrays on SiO_2 wafer and conventional photolithography. The increased friction force of this hierarchical hexagonal structure, especially in the hydrophilic state, demonstrated the importance of nanostructures on toe pads of tree frogs (figure 14b). Moreover, concave, spatular, T-shape and spherical micropillar tops were also constructed to investigate the adhesion properties. T-shape pillars, prepared by conventional moulding [59] and inking–printing–curing (IPC) methods, exhibited maximum adhesion strength, both in wet [60] and dry [48,61] conditions. Both the higher compliance and comparatively large contact area contribute to the outstanding performance. Furthermore, the crack initiation tends to start from the centre during the detachment [62], which results in a suction effect, contributing positively to the adhesion [59].

(b) Influences of micro- and nanostructure

Inspired by the existence of nanopillars in toe pad of tree frogs, we fabricated a composite structure with aligned polystyrene (PS) nanopillars embedded in soft PDMS hexagonal micropillars [63]. Both adhesion and friction forces were greatly enhanced compared to the structure with pure PDMS. The finite-element simulation suggests that the embedded nanopillars could regulate the stress distribution across the contact interface, with the stress maximum being distributed on top of a row of nanopillars close to the micropillar perimeter (but not at the centre of contact area or at the very edge of the pillar; figure 14d). This is similar to the T-shape pillars inspired by gecko setae, which can also shift the stress maximum to the centre of the contact area, inhibiting the crack initiation during pulling. However, the composite structure should possess a much higher stability and durability since the nanopillars are embedded.

Figure 14. (a) SEM images of hierarchical structure. (b) Plot of different microstructures on friction force. (c) Illustration of composite posts with an internal aligned-PS nanopillars. (d) Simulated stress distribution on composite micropillar during detachment. (e) Schematic of core-shell post with a stiff core and compliant shell. (f) Distribution of normal stress along the adhered interface of the post with $R_i/R = 5/6$. (a,b) Reproduced from Li *et al.* [57]; (e,f) are reproduced from Minsky & Turner [58].

More interestingly, this kind of stress-maximum shifting ability was also realized in a much simpler core-shell structure, which is composed of a rigid core of polyetheretherketone and a thin layer of PDMS [58,64] (figure 14e). It was found that a thinner layer of PDMS on the pillar end is more efficient in reducing the stress at the pillar edge, contributing to the adhesion enhancement. Similarly, by coating a thin layer of PDMS onto the gecko-inspired T-shaped micropillar made of polyurethane acrylate (PUA) could sharply enhance the shear adhesion as compared the same structure but composed of either PDMS or PUA [65]. If the core is composed of a polymer material whose stiffness significant decreases by applying an electric current, dynamic tuning of adhesion was successfully realized by the modulation of subsurface stiffness [66].

(c) Adhesion with liquid at interface

There is mucus on the toe pads of tree frogs, so that a thin layer of liquid is always present at the contact interface. Therefore, tree frog adhesion is considered as wet adhesion, compared to the dry adhesion of geckos. To mimic wet adhesion, spreading a layer of liquid on the structured surface is widely employed [60,67,68]. The wet adhesion strength is therefore profoundly influenced

Figure 15. (a) Adhesion force tested with different velocity on hydrophilic flat and structured surfaces. (b) Illustration of PDMS pillars coated with a responsive copolymer, realizing switchable adhesion under water. (a) Reproduced from Drotlef *et al.* [60]; (b) is reproduced from Ma *et al.* [71].

by the surface wettability [35,60]. By applying glycerol (which is polar and does not evaporate at room temperature) to the interface, the adhesion of structured PDMS against a ruby sphere was evaluated [60]. It was found that, in the main, short-range attractive forces dominate adhesion when the PDMS surface is hydrophobic, as the glycerol may be squeezed out from the contact area forming direct contacts. It therefore behaves like gecko-inspired structured adhesive, showing a strong dependence on the contact geometry [60]. By contrast, long-range attractive forces associated with capillarity dominate when the surface is hydrophilic. In this situation, capillary force dominates the adhesion strength, independent of the microstructures on the surface. It was suggested that the adhesion enhancement in the presence of fluid could be the result of crack arresting by liquid, in addition to the capillary forces [69]. It should be noted that excessive liquid will produce a lubrication region, leading to a reduction in adhesion and friction [69,70]. Since the surface wettability has a strong influence on wet adhesion, the switching of a surface between hydrophilic and hydrophobic, e.g. by temperature, could therefore regulate the wet adhesion. By coating a layer of temperature responsive copolymer poly(dopamine methacrylamide-co-methoxyethyl acrylate-co-isopropyl acrylamide) (p(DMA-co-MEA-coNIPAAm)) on PDMS micropillars, switching of underwater adhesion was successfully achieved by simply adjusting the temperature of the water bath (figure 15b) [71].

The microstructure of the toe pads of animals like tree frogs is considered to play a very important role in wet adhesion. It is suggested that the microchannels between pillars can effectively drain liquid out from the contact area, maximizing the effective solid–solid contact [55,67,72,73]. Due to the polygonal nature of frog-inspired micropatterns, shear adhesion (friction) shows orientation dependence. In our previous work, wet friction was found to be the highest along the direction of side-sliding (figure 16a) [55]. Moreover, elongated hexagonal micropillars show an even higher friction at the same orientation. The effective arresting of cracks increases following the edge density per unit length along the friction direction, contributing to friction enhancement. The theory was demonstrated by a macroscopic experiment, where the frog-inspired patterns were mounted on metal pieces and allowed to slide on a rotating surface covered with water (figure 16c). However, Cheng *et al.* reported a contradictory result that the wet friction of corner-sliding was higher in both hexagonal and rhomboid pillar arrays [74]. Using Chinese ink as the indicator, the liquid squeezing out of the contact interface was found to be more effective in the direction of corner-sliding (figure 16a). In the direction of side-sliding, liquid may flow into the contact interface due to the tilting of pillars and the flow pattern, resulting in decreased friction [74]. Recently, distinctive arch-shaped structures (figure 16b) were reported to provide even larger wet friction force than tree frog-inspired hexagonal patterns, due to their optimal drainage effect and the high stiffness of the patterns [75]. This offers new possibilities for the design of wet adhesives in future.

Recently, it was reported that the bioinspired wet adhesive exhibits better performance on rough than smooth substrates [76,77]. The introduced trace amounts of fluid result in capillary

Figure 16. (a) SEM image of hexagonal pillars with a different sliding direction for friction. Image on right shows the route for flowing water. (b) SEM images of different pattern (arch I, arch II and hexagon). (c) Image shows the angles at which flat and micro-patterned hydrophilic samples (regular and elongated hexagonal pillars) slide on a slant terrace flooded with water. (a) Reproduced from Chen *et al.* [74]; (b) Reproduced from Ko *et al.* [75]. (c) Reproduced from Iturri *et al.* [55].

bridges, raising the effective contact area [78] in a way analogous to a thin layer of soft material on top of micropillars [79]. This means that capillary force may serve as the major contributor to wet adhesion on a rough surface [77]. Moreover, structured adhesives could effectively slow down the evaporation of liquid on the rough substrates, which is quite important for tree frogs and other animals using wet adhesion in their natural environment [38].

While most studies of wet adhesion on artificial materials involved coating a thin layer of liquid on the structured surface, a few reported the delivery of liquid to the contact area mimicking the secretion of animals [49,80]. Making use of the microphase separation of block copolymer poly(styrene-*b*-2-vinylpyridine) (PS2VP), nanopillars with 98 nm internal channels were designed to deliver liquid to the contact area, mimicking the dynamic secretion of animals. The combination of material softening in high humidity and the transportation of mineral oil to the contact interface greatly increased the adhesion force by two orders of magnitude. The comparison of work of adhesion at different humidities suggests that the contribution of liquid bridges remained the same, while direct solid–solid contact was 30 times higher when the relative humidity increased from 25 to 90% (figure 17c). Interestingly, Vogel *et al.* [80] have designed a device with a plate full of holes. Water can be pumped into the holes via channels beneath the plate and form droplet arrays on the plate (figure 17a). Each droplet can therefore form a liquid bridge with a contacting surface, creating an adhesion force at the interface. Indeed, with all the liquid droplets acting together, the device can generate a substantial adhesion force.

4. Applications

Although the mimicking of tree frog-inspired wet adhesion is still in its infancy, there are already some attempts to make use of it in the area of bionic robots [81], soft tissue engineering [82], etc. For instance, Tsipenyuk prepared a variety of polyvinylsiloxane (PVS) hexagonal patterns as the stretching unit for disposable safety razors [82]. During the process of sliding against lubricated

Figure 17. (a) Image shows large number of liquid bridges were quickly formed by electronic control. (b) SEM image of continuous porous pillars. (c) Dependence of $W_{ad,S}$ and $W_{ad,C}$ on F_L at RH about 90%. (c) Reproduced from Vogel *et al.* [80].

Figure 18. (a) Schematic illustration of the preparation process of the commercial safety razor cartridge with hexagonal surface. (b) Pig liver was deformed by using a normal force of 10 N. (c) Schematic illustration of bioinspired patch for detecting ECG signals. (a) Reproduced from Tsipenyuk *et al.* [82]. (b) Reproduced from Chen *et al.* [74]. (c) Reproduced from Kim *et al.* [83].

human skin, the hexagonal surface could increase the effective contact with the skin by draining excessive liquid through the channels. Thus, the friction force was double that of commercial products and stretched the moist skin much better. Also, Chen *et al.* prepared PDMS polygonal arrays and applied them to the gripping surface of surgical graspers (figure 18b). Compared to the traditional tooth-textured surgical grasper, the new design can greatly reduce the gripping force, reducing damage to soft tissue [74]. More recently, a biomimetic skin patch inspired by the hexagonal pattern of tree frog toe pads and the convex cup of octopus suckers has been reported (figure 18c) [83]. The patch exhibited outstanding adhesion performance on human skin, even in

a water flowing environment. In addition, following coating with reduced graphene oxide, the patch could serve as a flexible electrode, sensitive enough to receive bio-signals on a wet skin, even under motion.

5. Conclusion and outlook

It is clear that tree frogs exhibit outstanding wet adhesion properties. Significant progress has been made in recent years, both in our understanding of how tree frogs adhere and in the development of tree frog-inspired wet adhesives. But many questions remain unanswered. For instance, while most of the tree frog-mimicking studies have focused on the micropattern on the toe pad, more attention should be given to forming a better understanding of the role of the nanostructures. Could there be synergistic interactions between the micro- and nanostructures and the mucus? Moreover, since the animals keep secreting all the time, the liquid volume at the contacting interface should change accordingly. How will this dynamic process contribute to adhesion and friction during the locomotion of animals? And can we make use of the dynamic process? The answers to these questions could deepen our understanding of the adhesion of tree frogs and speed the development of applications of bioinspired wet adhesion.

Data accessibility. The article has no additional data.

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Competing interests. The authors declare that they have no competing interests.

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