The wetting and pagoda instability (PI) on soluble fibers

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We discover a new kind of instability when inserting a soluble fiber into liquid. Because the dissolved fiber looks like a Chinese pagoda, we designate this instability as pagoda instability (PI). During the process of PI, coupling of dissolution and wetting leads to special phenomena, i.e. periodic instability, dissolving-induced jet flow, etc., and optimizes the fiber shape, which can be used to screen the capillary force. In this paper, we study PI of moving contact line on the surface of soluble fibers, obtain a phase diagram of the different shapes of the dissolved fibers, reveal evolution dynamics and mechanisms of the interface and the energy influenced by the competition between interface energy and chemical potential, and propose a criterion of PI to predict the occurrence of PI and shape of the dissolved fibers. At the end of this paper, to show the important application of PI, we design and prepare a new AFM tip, which can shield the precision of AFM measurement from the capillary effect caused by ambient humidity, liquid film, etc.
The study on the behaviors and mechanisms of wetting on insoluble fibers can be traced back to the Goucher and Wards’ work about liquid film\(^1\), and its critical importance in the field of directional droplet transport\(^2\), coating\(^3\), surface modification of fibers\(^4\), preparation of micro-/nano-devices\(^5\), etc. Through decades of efforts, a clear physical picture of wetting on fibers has been achieved. When a lyophilic fiber is inserted into liquid, the surface tension drives the liquid to climb along the fiber. The liquid keeps rising until the wetting system reaches a steady state, when the gravitational force is balanced by the interface tensions. The final height of moving contact line (MCL) \(H \sim R \ln \left(2 \kappa^{-1}/R\right)\), where \(R\) is the fiber radius, \(\kappa^{-1} = \sqrt{\gamma / \rho g}\) the capillary length, \(\rho\) the density, and \(g\) the gravitational acceleration\(^6\). Once the fiber is pulled out from the liquid, Plateau-Rayleigh instability (PRI) happens. The liquid film on the fiber surface breaks into periodically-distributed droplets. The distance between neighboring droplets is proportional to the initial radius \((R_i = h_i + R, \text{ where } h_i \text{ is thickness of the initial liquid film})\). PRI is dominated by surface tension, which minimizes the surface area of the liquid and the surface energy of the wetting system.

However, when we insert a soluble fiber into liquid, new phenomena and mechanisms immerge, which cannot be explained by the previous theory of wetting on insoluble fiber. At first, the liquid rises along and dissolves the fiber surface. The maximum height of the MCL is no longer proportional to \(R_i\), but is determined by both of \(R\) and the contact angle \(\theta\) which is between solid-liquid (SL) and vapor \(^7\). Once the MCL reaches its final height, a new kind of interfacial instability occurs, the liquid falls quickly along the fiber surface (Fig. 1). This new instability, namely the “pagoda instability (PI)”, is the product of the coupling effect of dissolution and wetting, and is also the result of the competition between chemical potential and interfacial energy. PI optimizes the shape of the soluble fibers to be a special pagoda shape, which can greatly reduce the capillary force between the liquid and the fiber, and make the capillary rise hardly occur.

The PI-optimized tips are capable of low adhesion, high penetration, screening capillary effect, etc., therefore related technologies are gradually used to optimize and design structures and materials with such requirements. However, the theory of wetting on insoluble fibers are no longer applicable to the wetting and PI on soluble fibers, which becomes the bottleneck in the application fields, such as the tip of optical sensor \(^8\), low-adhesion particles mimicking pollens \(^9\), micro-needle patches for rapid and painless injection of drugs \(^10\); spiky particles enhancing humoral antigen
specificity and cellular immune response, super-hydrophobic surfaces that easily recover Cassie state, and so on. Therefore, it is necessary to carry out systematically and depth studies on the dynamics and mechanisms of MCL on soluble fibers.

In this paper, we discover a new “pagoda instability” when inserting soluble fibers into liquid. We design a series of experiments to quantitatively investigate the influence of the physical/chemical properties and the initial shape of fibers on the interfacial behaviors, explain the dissolving-induced jet flow, interpret the coupling of the fiber dissolution and capillary rise, clarify the reasons why the fibers dissolve into different shapes, reveal the dynamic rules and mechanisms of the evolution of interface and energy, as well as obtain the criterion of PI. Based on our results, we can predict the occurrence of PI and the final shape of the dissolved fiber. At the end of this paper, to demonstrate the practical applications of PI, new AFM tips were designed and prepared. Compared with the commercial AFM tips, our PI-optimized tips can effectively reduce the capillary effect on the AFM measurement.

Our investigated objects are fibers with different radiuses (0.2mm~5mm), and then we found the fibers evolved into various structures gradually (see Fig. 2). When the water rises along the fiber wall to a certain height, the water-oil interface bends to glucose-water interface and touches with fiber wall. At last, the water-oil interface falls to a new position, and then it rises again. This loop repeats until the water-oil interface separates from fiber wall (see Fig. 1a). Every process of the rise and fall is similar, so the shapes of each floor on the surface of fiber are similar. According to results of experiments, we propose that the forming process of pagoda structure on the fiber wall is self-similar. Therefore, if we clarify the forming mechanism of the first floor, we will deduce the whole process of fiber dissolving.

The profile of the first floor is shown in Fig. 3(a). Without loss of generality, we build a model in which the fiber is surrounded by two liquids. One is solvent (the symbol is L), the other is covering that is immiscible with solute and solvent (the symbol is O). If the outside factors can be ignored, the covering does not need in the experiments. Then, the symbol O represents air. Disgusting with the dissolution in quiescent liquid, the dissolution in triple-phase contact line results in instability. The forming process of pagoda structure can be divided into three stages: liquid rise, interface instability, and liquid fall.

Unbalanced forces cause the liquid to rise. We analyze the driving forces and resistances in the
contact line, which arise from free energy and dissipation respectively. Free energy consists of interface energy and chemical potential results from dissolution. With the change of interface shape induced by dissolution, the direction of surface tensions and the interfaces vary, and then interface energies change. It is noted that the influence of concentration to surface tensions is so light that can be ignored based on the results of experiments\textsuperscript{14}. When the solute enters the liquid from solid, solvent molecules surround it. Therefore, the chemical potential is related with the construction of solute molecules except the concentration. We use soluble energy density $\Gamma$ to characterize the solute influence to solution\textsuperscript{15}. The dissolution effect can be expressed as special surface tension $2\Gamma \tilde{\sigma}_{Sl}$, where $\tilde{\sigma}$ and $\delta_{Sl}$ are boundary layer thickness near interface $SL$ and averaged concentration in boundary layer, respectively. The dissipation is induced by contact line “friction” and liquid viscosity \textsuperscript{16}. The contact friction can be express as $\pi \tilde{\xi}_{St} R \dot{h}$, where $\tilde{\xi}_{St}$ is friction parameter, $R_{n}$ is the $n$th floor initial radius, $\dot{h}$ is the liquid rise velocity. The viscosity dissipation mainly exists in the boundary layers of liquid L and liquid O. The viscosity dissipation is expressed as $3\eta R \tilde{\sigma}^{-1} \int_{0}^{\dot{h}} v^{2} dz$, where $\eta$ is liquid viscosity, $\tilde{\sigma}$ is boundary layer thickness, $v$ is flow velocity near liquid L-liquid O (LO). The boundary layer thickness is proportional to $\sqrt{\eta \kappa^{-1} / \rho L v}$ where $\rho_{L}$ is the density of liquid L. Besides, the flow velocity is close to the rise velocity $\dot{h}$, i.e. $v \sim \dot{h}$ in this zone. Given that the characteristic length of the zone is lower than capillary length, we ignore the gravity effect. Because the rise velocity is slow, we hypothesize the process of rise is quasi-static. In other words, the driving forces always keep balance with resistances. Consequently, based on Onsager’s variational principle and ignoring the change of capillary number\textsuperscript{17}, we obtain the scaling law of liquid rise

$$ h \propto t^{1/3} \tag{1} $$

The capillary number can be expressed as $Ca = \eta \dot{h} / (\gamma_{Sl} - \gamma_{SL} \cos \theta_{SL} + \gamma_{LO} \cos \theta_{LO} + 2\Gamma \tilde{\sigma}_{Sl})$, where $\gamma$, and $\theta$ are surface and tension, contact angle, respectively. The capillary number shows that the contact angles are related to the rise velocity. Liquid rises with the constant advancing angle\textsuperscript{18} that is the sum of $\theta_{SL}$ and $\theta_{LO}$. With the increasing of contact angle $\theta_{SL}$ due to the
recession of interface $SL$, the contact angle $\theta_{LO}$ decreases, and the rise velocity of the liquid slows down. It is noted that the contact angle $\theta_{LO}$ we mentioned is apparent contact angle, and the liquid in meniscus is in equilibrium with the liquid bath because of the quasi-static hypothesis. The change of liquid height with time cannot reflect the evolution of interfaces in detail unless the function of interface evolution is obtained. Given that overpressure equals to zero owing to the equilibrium of liquid in meniscus and liquid bath, the profile of interface $LO$ satisfies the equation $\frac{f_{LO}}{\sqrt{1+(df_{LO}/dz)^2}} = R_s$. The condition $df_{LO}/dz|_{z=0} = \tan \theta_{LO}$ leads to the function of interface $LO$. We ignore the gravity effect on the interface $LO$ based on the low Bond number $Bo = \frac{\rho_s R^2 L}{\eta}$.  

The liquid rises accompany by the recession of the interface $SL$. As shown in Fig. 3(b), the dissolution takes place in boundary layers near the interface $SL$. The thickness of boundary layer depends on viscosity $\eta$ and diffusivity $D$. The relative importance of the viscosity and the diffusivity can be characterized by Schmidt number $Sc = \frac{\eta}{\rho L D}$, and we estimate $Sc \in 10^3$ in most cases. Therefore, high Schmidt number implies that solid dissolves into solvent by shear effect rather than diffusion. Thus, the dissolution velocity of radial direction depends on the shear stress in boundary layer $\delta_{SL}$, i.e. $v_s \propto \frac{\tau}{\delta_{SL}}$, where $v_s$ is the flow velocity near the interface $SL$. We ignore the influence of Marangoni convection to the flow velocity near the interface $SL$. Because the characteristic velocity of the Marangoni convection $u_m = \frac{\gamma_c L}{\eta} \approx 10^{-2}$ m/s is much lower than that of the nature convection $u_s = \frac{\Delta \rho g L^2}{\eta} \approx 1$ m/s. The velocity can be calculated by considering buoyancy $\Delta \rho gL^2$ and viscous force $\eta v_s L$, where $\Delta \rho = |\rho_s - \rho_l|$, $\rho_s$ is the density of solute. $L_c (L_c \sim R \sim 10^{-3}$ m) is the characteristic thickness of the liquid L on the fiber, and the velocity can be further expressed as $v_s \propto \frac{\sqrt{\rho_l z}}{R}$. The function can describe the results of raw data well (Fig. 4b). Because of the slow flow velocity, we think that the solute particle and solvent particle have the same velocity $v_s$. It is noted that the flow velocity closed to contact line is not zero and equals to $h$. We disregard it in the expression of $v_s$ because the velocity is too low compared with the velocity in origin. The boundary layer thickness near interface $SL$
\[
\delta_{SL} \propto \sqrt{\eta L \left(1 - \frac{z}{h}\right)^7 / \left(\rho_c v_y\right)},
\]
where \(\rho_c\) is the characteristic density of liquid in this boundary layer. Thus, when \(z > 0\), the scaling law of dissolution velocity in radial direction can be expressed as \(v_u \propto (h - z)/R^2\). As we said before, interface SL recedes with the rise of liquid. Thus, the dissolution time is dominated by liquid rise, then we can obtain dissolution time is \(t_d \propto \left(h^3 - z^3\right)\), according to the Eq. (1). Obviously, the profile of the interface SL can be represented as \(f_{SL} = v_n t_d\).

It is worth to explain that the dissolution in the zone below the horizon is different, because the flow separation occurs at the end of fiber. The flow velocity slows down. We assume the dissolution of solid below the horizon is uniform, because the slope of fiber wall is small (\(<5^\circ\)). Therefore, we think the shape of fiber below the horizon keeps cylindrical, and the dissolution velocity in radial direction and dissolution time satisfy \(v_u \propto h/R^2\) and \(t_d \propto h^3\), respectively. There is quiescent region on the bottom of fiber because the flow separates from the end of fiber. In the quiescent region, the flow velocity is very slow and about \(10^{-4} \text{ m/s}\). The transport method of solute depends on the relative importance of convection velocity (or flow velocity) and diffusion velocity, whose ratio is Péclet number. As we mentioned before, the diffusivity \(D\) is about \(10^{-9} \text{ m}^2/\text{s}\), and the size of quiescent region \(\delta_q \propto R\), and its scale is \(5 \times 10^{-4}\), approximately, by experiment measurement. Obviously, Péclet number is \(10^{-2}\) and so low that the convection effect can be ignored in quiescent region. The change of the fiber length can be expressed as \(\Delta l = D(c_j - c')t_d / \delta_q\). According the Fig. 1(a) of tip forming, the key of pagoda construction formation is liquid fall caused by instability. Therefore, we further study the process of interface instability.

The Fig. 5(a) shows the instability of interface is the key reason of pagoda structure forming. According to the results of experiments (see Fig. 5), The instability is much different from Plateau–Rayleigh instability. Therefore, we think it is a new instability. From an energy perspective, the liquid rise is a process of free energy desecration. For the rise without dissolution, the interface SL replaces the interface SO whose interface energy is higher. During the rise of liquid on the surface of soluble fiber, liquid rise accompanies dissolution. The interface SL replaces the interface SO while solute molecules enter the solvent further reduce the free energy. As a result, the height of liquid rise accompanied dissolution is higher than that without dissolution. According to the above
analyses, we built a model to calculate free energy of the system $F$ which is the function of liquid height $h$ and includes the change of interface energy and chemical potential of solution. The increase of interface $SL$ energy change $\gamma_{SL} \Delta A_{SL}$ always accompanies the decrease of interface $SO$ energy $\gamma_{SO} \Delta A_{SO}$. Similarity, the change of interface energy in the interface $LO$ can be written as the difference between the initial state $\gamma_{LO} \pi \left( f_{LO} \right)_{h=0}^{\infty}$ and the instant state, i.e. $\gamma_{LO} \Delta A_{LO}$. As we said before, the dissolution occurs in the boundary layer $\delta_{SL}$ near the interface $SL$, the concentration of solution beyond the boundary layer $\delta_{SL}$ approaches zero. The average volume concentration in boundary layer $\bar{\delta}_{SL}$ is $\bar{\tau} = \nu_s / \nu_g$. Thus, the change of chemical potential in solution resulting from dissolution is $\gamma_{SL} \Delta V_{SL}$, where $\Delta V_{SL}$ is the volume change of boundary layer. The change of free energy is calculated by numerical method and shown in Fig. 6(a) by red lines for different stages. Obviously, with the rise of liquid along the fiber, the free energy has the minimum value, i.e. potential well. Elaborating on this process, the free energy decreases because the area of the interface $SL$ increases resulting from liquid rise and dissolution. The dissolution keeps proceeding because the solution is unsaturated. The perturbation makes liquid oscillates around the potential well (\( dF/dh = 0 \), \( d^2F/dh^2 > 0 \)). We further calculate the change of free energy during the liquid fall whose results is shown in Fig. 6(a) by blue lines. We find that the liquid fall can make the system reaching to a more stable state. Besides, the gradient of blue line is higher than that of red line in the intersection of two lines. The system reaches the more stable state along the way with higher gradient. Given that, the position of potential well is not the stable point of the system; the liquid cannot stay in the position for a long time. On the liquid’s way back to the position of potential well, the liquid keeps falling rather than staying in the position of potential well. We call this process as loss of stability. As shown in the Fig. 5(a), during the loss of stability, the interface $LO$ keeps bending until to touch the interface $SL$. At the same time, the results of free energy show that the liquid does not fall to the origin and stop in a certain position that we call it as recovery of stability. It is noted that the liquid falls with the receding angle that distinguishes with advancing angle. In the process of liquid fall, the fall velocity is much higher than dissolution velocity, so we disregard the dissolution during liquid fall. The contact angle $\theta_{SL}$ approaches zero, and the receding angle is
lower than advancing angle. During liquid L dissolving the solid, the interface $LO$ loss stability as soon as liquid L reaches a certain height $H_n$, which is height of the $n^{th}$ floor. By calculating the null point of derivative of the function $F$ that the second derivative of $F$ greater than zero, we can obtain the criterion of the interface instability

$$\frac{H_n^3}{L_{SL} R_n^2} \propto \frac{\varphi_c + 2\gamma_{SO}}{\varphi_c + \gamma_{SL}},$$

(2)

where the symbol $\varphi_c = \delta_{SL} \rho_s N_A \Gamma V_{SL} \tau / M_s$ is the dissolution energy per area. $M_s$ is the mole mass of solute. $L_{SL} = C^3 \sqrt{\eta / \left(4C_r u_n^{1/2} \sqrt{\rho \tau} \right)}$ is the characteristic length of interface $SL$, and the $C_r$ and $C_t$ are constant. The $C_t = h / l^{1/3}$, and the $C_r = v_a / \tau$. The expression of criterion is a power function because of the self-similarity instability process. Consequently, when the scaling law is $2/3$, i.e. $H_n \propto R_n^{2/3}$.

The Fig. 6(a) shows the falling of liquid can further decrease the free energy of the system, and we disregard the dissolution effect during liquid falling, because the falling velocity is much quicker than the dissolution velocity. Therefore, the height of stability recovery can be expressed as

$$J_n \propto R_n^{-1} \ln \frac{2k^{-1}}{R_n^{-1}},$$

(3)

which is similar with the height of the meniscus on the insoluble surface. The curves in Fig. 6(a) also show that the PI is periodic, that is the reason why solvent erodes the fiber into pagoda structures. According to the Fig. 6(a), the variations of free energy in each instability are similar, and that further proves that the fiber erosion is a self-similarity process.

At the beginning of article, we mention that the fiber can be eroded into different shapes including single floor, pagoda-like, cuspidal tip, and flat end. Here, we elaborate on the forming process of tip. During the formation of the tip, the dissolution velocities on the fiber wall (radial direction) $V_r$ and on the bottom of fiber (axial direction) $V_a$ compete with each other. As we mentioned before, the convection effect dominates the dissolution on the fiber wall, and the diffusion effect dominates the dissolution on the bottom of fiber. Thus, in general, the dissolution velocity of axial direction is slower than radial direction. Unless the insertion depth of fiber inserted is shallow, the fiber will
format a cuspidal tip. When the liquid separates from the fiber, or the dissolution depth in the radial direction is greater than the radius of fiber during the first instability, the fiber will format a single floor. Because of the axial dissolution of fiber, the inserted depth \( l \) is the key parameter that decides the shape of tip. The maximum dissolution velocity of radial direction is proportional to \( H^{3/4} \) that is decided by \( R_1 \), the radius \( R_1 \) is another key parameter that decides the shape of tip.

Besides, the radius \( R_1 \) also decides the position of stability recovery \( J_1 \). According to the parameters \( l \) and \( R_1 \), we can divide all sizes of fibers into four types and draw the boundary of every region in phase diagram. As we depict in the Fig. 2(e), for example, the fibers are included in the third zone that have single floor with cuspidal tip.

As we known, screening the capillary force is vital to scan the sample that has interface \( SL \) by AFM. If the depth of liquid over 200 \( \mu \)m, the tip is completely immersed in liquid to avoid the influence of liquid-air interface. However, when experiments are proceed in ambient humidity or on the surface of samples with liquid film, there is a liquid bridge between usual tips and sample owning to liquid adhesion. The capillary pressure induced by liquid bridge will decrease the measurement accuracy of the usual tip. Using The PI-optimized tips can greatly improve the precision of experiment, because the special shape of tip helps to prevent the rise of liquid. The results of the AFM experiment are shown in Fig. 7.

In summary, we observe and explain the dissolution process of the fiber inserted into solvent. The liquid rise along the soluble fiber has a new scaling law 1/3. The experiments show that the instability during dissolution is the key reason to the formation of fiber shape. Based on the shape of interface \( SL \) and interface \( LO \), we calculate the free energy of the system and find the positions of stability loss and stability recovery. Consequently, we propose the criterion to describe the instability. Besides, the change of free energy further proves and explains that the formation of fiber shape is self-similarity. According to the results of scaling analysis and experiments, we find four types tips that can be obtained by dissolving different sizes fiber. We elaborate that by phase diagram.

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Fig. 1 | The process of pagoda instability. a, the Time-lapse photography of the process of the pagoda instability; b, Chinese pagoda.
Fig. 2 | Shapes of a dissolving fiber. Fibers made of glucose were inserted into water, and silicone oil was used as covering layer. Candy fibers (dark areas) can be dissolved into four types: a, pagoda-like with flat end; b, pagoda-like with cuspidal tip; c, single floor with cuspidal tip; d, single floor with flat end. e, Diagram shows the geometrical conditions obtaining different shapes fibers that include pagoda-likes with flat end, pagoda-likes with cuspidal tip, single floor with cuspidal tip, single floor with flat end (light gray, light yellow, light blue and light green, respectively.).
**Fig. 3 | Schematics of the model.** a, Liquid rises along the soluble fiber while liquid L dissolves solid. When the liquid rises to the certain position $H_n$, the interface LO bends to fiber. c, After instability, liquid L falls to new position. At last, the liquid rises again and enter next loop until the liquid separates with fiber.
**Fig. 4** Flow field. 

**a.** The low concentration liquid flows up along Water-Silicone oil interface and dissolves the glucose. The high concentration liquid containing glucose flows down while the interface SL recedes. 

**b.** Plots shows that the velocity near the interface SL $v_g$ is proportion to $h$ and inversely proportional to $R$. 
Fig. 5 | Interface evolution of a dissolving fiber. 

a, The time-lapse photos show the change of interface. Red lines are interfaces $SL$, and blue lines are oil-liquid interface. 

b, Plot shows the change of rise height $h$ with time. $t_d$ is the time taking to rise. 

c, PIV experiment indicate that the quiescent region exists in the bottom of fiber.
Fig. 6 | Energy evolution and criterion of pagoda instability. a, The diagram of free energy shows the change of energy in every loop (one rise and one fall) is similar. b, the instability criterion presents the scaling law of $H_n$ changing with $R_n$ is 0.5.
Fig. 7 | Measuring adhesion force by AFM. The black and pink lines are the loading curve of optimized tip and usual tip, respectively. The blue and green lines are the uploading curve of optimized tip and usual tip, respectively.