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Capillary pumping independent of liquid sample viscosity

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Abstract

Capillary flow is a dominating liquid transport phenomenon on the micro- and nanoscale. As described at the beginning of the 20th century, the flow rate during imbibition of a horizontal capillary tube follows the Washburn equation, i.e. decreases over time and depends on the viscosity of the sample. This poses a problem for capillary driven systems that rely on a predictable flow rate and where the liquid viscosity is not precisely known. Here we introduce and successfully experimentally verify the first compact capillary pump design with a flow rate constant in time and independent of the liquid viscosity that can operate over an extended period of time. We also present a detailed theoretical model for gravitation independent capillary filling, which predicts the novel pump performance to within measurement error margins, and in which we, for the first time, explicitly identify gas inertia dominated flow as a fourth distinct flow regime in capillary pumping. These results are of potential interest for a multitude of applications and we expect our results to find most immediate applications within lab-on-a-chip systems and diagnostic devices.

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Introduction

Capillary pumping is utilized in many applications, e.g. for manipulating liquids in heat pipes¹, for regulating fluid flow in low-gravity environment in space², for patterning biomolecules on surfaces³, as pumping mechanism in immunoassays⁴, and in diagnostic applications⁵. In many of these applications, precise control over flow rates is of fundamental importance to their function. This is especially evident for immunoassays and other diagnostic applications, where the biosensor signal depends on a chemical reaction that is directly affected by the transport of the sample in the reaction zone⁶. In these biomedical applications, samples of interest, e.g. urine and blood, show large sample-to-sample variations in viscosity: the relative standard deviation in healthy individuals is 10% and 33%, respectively⁷, with even larger deviation occurring in many health conditions (e.g. $\sim 4\times$ viscosity increase for polycythaemia). Additionally, whole blood behaves viscoelastically. Since the capillary flow rate described by the Washburn equation depends on the viscosity of the sample and decreases over time⁸⁻¹⁰, precise control over the assay conditions in capillary driven systems remains an unresolved challenge.

Four flow regimes can be discerned during capillary filling, which are, subsequently: gas inertia dominated flow (explicitly identified for the first time in this work); liquid inertia dominated flow; gas viscosity dominated flow and liquid viscosity dominated flow. In most capillary pump configurations, the first three flow regimes only occur during a negligibly short period of the capillary filling process. Therefore, liquid viscosity dominated flow, i.e. where the influence of the displaced fluid is negligible and where the fluid inertia is negligible, has been most extensively studied, and was described in three papers during the period 1905-1921 by Bell, Cameron, Lucas and Washburn⁸⁻¹⁰. Liquid viscosity dominated flow is characterised by the liquid front position, $\mathbf{x}$, moving inversely with the square root of time, t : $d\mathbf{x}/dt \sim t^{-0.5}$. In 1923, Bosanquet¹¹ was the first to provide a theoretical model encompassing both the effects of the imbibing and displaced fluids. In 1997, Quéré was the first to experimentally study the liquid inertia dominated flow regime, which is characterised by a constant liquid

front velocity: $dx/dt \sim t^0$.¹² In 2011, the influence of gas as the displaced fluid, particularly the gas viscosity dominated regime, was first studied in detail by Hultmark et al.¹³ In their experiments, and despite using very long capillaries, they observed gas viscosity dominated flow only during the first 0.5 s of capillary filling, i.e. a too short time span for practical applications such as point-of-care diagnostics.

In this work, we start with providing a more detailed theoretical derivation of the flow regimes. Herein, we explicitly identify for the first time the gas inertia dominated flow regime as a fourth flow regime. Thereafter we introduce a compact capillary pump design that operates in the gas viscosity dominated regime, i.e. with flow rate constant in time and independent of liquid viscosity, that can operate for several tens of minutes at a relevant flow velocity. Briefly, this is achieved by adding a narrow air vent downstream in the capillary system, which ensures that the viscosity of the displaced fluid, air, dominates over the viscosity and inertia of the imbibing liquid, as illustrated in Fig. 1. We study the potential operational regime of the new design in terms of volumetric flow rate and pump velocity. Finally, we demonstrate the concept experimentally.

Figure 1 Design of our novel capillary pump. a) In constant cross-sectional area capillary tubes, liquid filling follows the Washburn behaviour during most of the imbibition, resulting in a flow rate that decreases over time and depends on the viscosity of the sample. b) The capillary pump design introduced in this paper features a narrow downstream air vent, which ensures that the viscosity of the displaced air in the vent dominates over the viscosity and inertia of the imbibing liquid. This results in capillary filling with a flow rate independent of time and liquid viscosity.

Theory

We study liquid imbibition of horizontal capillary tubes with constant cross-section, in which air is the displaced fluid. Considering a Laplace pressure drop driven laminar Poiseuille flow, the regimes can be described as follows. When liquid enters a long capillary tube at $t = 0$, a stationary liquid-gas curved interface establishes during an initial acceleration in the region $x < r$. Once a stationary gas-liquid interface is established, the equation of motion is, neglecting gas compression,

$$P_c = \frac{d}{dt}[(\rho_L \cdot \mathbf{x} + \rho_G \cdot (l - \mathbf{x})) \cdot \frac{d\mathbf{x}}{dt}] + g \cdot (\mu_L \cdot \mathbf{x} + \mu_G \cdot (l - \mathbf{x})) \cdot \frac{d\mathbf{x}}{dt}, \quad (1)$$

where l is the length of the capillary tube, μ_L and ρ_L are the viscosity and density of the liquid, respectively, μ_G and ρ_G are the viscosity and density of the gas, respectively, P_c is the capillary pressure, and g is a constant depending only on the cross-sectional geometry of the tube. For tubes with circular cross-section with radius r , $P_c = \frac{2\gamma\cos(\theta)}{r}$ and $g = 8/r^2$, where γ is the liquid-gas surface energy and θ is the contact angle between the liquid and the tube wall.

In dimensionless form ($\mu = \mu_L - \mu_G$, $\mu^* = \mu_G/\mu$, $\rho = \rho_L - \rho_G$, $\rho^* = \rho_G/\rho$, and $\mathbf{x}^* = \mathbf{x}/l$), integrating over time, and posing boundary conditions $\mathbf{x} = 0$ and $u = \frac{d\mathbf{x}}{dt} = 0$ at $t = 0$, we obtain

$$\frac{P_c}{l^2} \cdot t = \rho \cdot (\mathbf{x}^* + \rho^*) \cdot \frac{d\mathbf{x}}{dt} + g \cdot \mu \cdot \mathbf{x}^* \cdot \left(\frac{\mathbf{x}^*}{2} + \mu^*\right). \quad (2)$$

This equation allows identifying the four flow regimes. When the inertia term dominates in the right hand expression of eq. 2, it simplifies to

$$\frac{P_c}{l^2} \cdot t = \rho(\mathbf{x}^* + \rho^*) \frac{d\mathbf{x}}{dt}, \quad (3)$$

which allows solving

$$\mathbf{x}^* = \sqrt{\rho^{*2} + k_{inert}^2 \cdot t^2} - \rho^*, \quad (4)$$

Figure 2 Theoretical model and capillary pump design. a) The theoretical dimensionless liquid front velocities u_{visc}^* (red) and u_{inert}^* (green) at different dimensionless liquid front positions x^* during the imbibition of a capillary tube where all four flow regimes dominate in some part of the tube. Note that $>98\%$ of the capillary pump fills with a liquid viscosity dominated flow regime. b) The theoretical dimensionless liquid front velocities u_{visc}^* (black, red, orange) and u_{inert}^* (green) of liquids of varying viscosity during the imbibition of the viscosity independent capillary design. Inset c) shows a close-up of the capillary positions $r < x < l_p$, highlighting that the velocity is nearly independent of the liquid viscosity ($\delta < 0.1\%$). d) Illustration of the experimentally tested compact capillary pump design, for which the theoretical behaviour in section $r < x < l_p$ is predicted as in graphs b and c. Note that $>99.4\%$ of the capillary pump section $0 < x < l_p$ fills with a liquid viscosity dominated flow regime.

and

$$\begin{aligned} u_{inert} &= \frac{d\mathbf{x}^*}{dt} = k_{inert}^2 \cdot t \cdot [\rho^{*2} + k_{inert}^2 \cdot t^2]^{-1/2} \\ &= k_{inert} \cdot \sqrt{1 - \left(\frac{\rho^*}{\mathbf{x}^* + \rho^*}\right)^2}, \end{aligned} \quad (5)$$

where $k_{inert} = \sqrt{\frac{P_c}{\rho \cdot l^2}}$.

For $\mathbf{x}^* \ll \rho^*$ one finds the gas inertia dominated regime, where

$$u_{inert} \approx k_{inert} \cdot \sqrt{\frac{2 \cdot \mathbf{x}^*}{\rho^*}}. \quad (6)$$

For $\mathbf{x}^* \gg \rho^*$ one finds the liquid inertia dominated regime studied by Quéré¹², where

$$u_{inert} \approx k_{inert}. \quad (7)$$

When the viscous term dominates in the right hand expression of eq. 2, it simplifies to

$$\frac{P_c}{g \cdot \mu \cdot l^2} \cdot t = \mathbf{x}^* \cdot \left(\frac{\mathbf{x}^*}{2} + \mu^*\right), \quad (8)$$

which allows solving

$$\mathbf{x}^* = \sqrt{\mu^{*2} + 2 \cdot \mu^* \cdot k_{visc} \cdot t} - \mu^* \quad (9)$$

and

$$u_{visc} = \frac{d\mathbf{x}^*}{dt} = k_{visc} \cdot [\mu^{*2} + 2 \cdot \mu^* \cdot k_{visc} \cdot t]^{-1/2} = k_{visc} \cdot \frac{\mu^*}{\mathbf{x}^* + \mu^*}, \quad (10)$$

where $k_{visc} = \frac{P_c}{g \cdot \mu_G \cdot l^2}$.

For $\mathbf{x}^* \ll \mu^*$ one finds the gas viscosity dominated regime, during which

$$u_{visc} \approx k_{visc} \cdot \left(1 - \frac{\mathbf{x}^*}{\mu^*}\right). \quad (11)$$

For $\mathbf{x}^* \gg \mu^*$ one finds the well-known Lucas-Washburn regime, dominated by liquid vis-

cosity, where

$$u_{visc} \approx k_{visc} \cdot \frac{\mu^*}{\mathbf{x}^*}. \quad (12)$$

Fig. 2a plots the dimensionless velocities $u_{visc}^* = u_{visc}/k_{visc}$ and $u_{inert}^* = u_{inert}/k_{visc}$ against $\mathbf{x}^*$ for a capillary system design ($r = 180 \mu\text{m}$; $l = 30 \text{ cm}$; using water and air as fluids and assuming an equilibrium contact angle $\theta = 0$) where all four regimes are dominating over some interval of x . Note that it is not generally the case that all four flow regimes dominate over some interval of x : our novel capillary pump introduced in this manuscript, for example, is designed to not feature inertia dominated regimes.

Design

A pump designed to operate in the gas viscosity dominated regime for values of $\mathbf{x}^* \rightarrow 0$ would feature a nearly constant flow velocity that is independent of the liquid viscosity. Indeed, for a first section of length l_P of such capillary pump, where l_P is chosen such that

$$l_P^* = l_P/l \ll \mu^*, \quad (13)$$

substituting $\mathbf{x}^* = l_P^*$ in eq. 11 shows that the dimensionless liquid front velocity variation would be less than a factor $\delta = l_P^*/\mu^*$:

$$1 > u_{visc}^* > 1 - l_P^*/\mu^* = 1 - \delta. \quad (14)$$

To design such capillary pump with a relevant (long enough) gas viscosity dominated pump section l_P requires expanding the region where the flow is gas viscosity dominated: gas viscosity domination should start very close to the inlet of the capillary, ideally at a position $x < r$; and the start of the liquid viscosity dominated region should be moved far enough downstream. The first requirement can be accomplished by designing the system

such that

$$\forall \mathbf{x} > r, \quad u_{inert} \gg u_{visc} \Leftrightarrow \frac{l}{r^2} \gg \frac{\rho_G \cdot \gamma \cdot \cos(\theta)}{64 \cdot \mu_G^2}, \quad (15)$$

where the right hand inequality follows from substituting eq. 6 and $u_{visc} \approx k_{visc}$ in the left hand inequality and solving for $\mathbf{x}^* = r/l$.

The second requirement can be accomplished by a long enough capillary length l : indeed, eq. 14 shows that it suffices to choose

$$l > l_P / \delta \mu^*, \quad (16)$$

to achieve any arbitrarily small relative velocity change δ .

Choosing l and r to fulfill eqs. 15 and 16 thus ensures a capillary pump that is gas viscosity dominated, i.e. with constant and liquid viscosity independent flow rate, over a length interval $x \in [r \quad l_P]$.

Fig. 2b-c plots the calculated liquid front velocity for such gas viscosity dominated capillary with $r = 0.7$ mm, $l = 6600$ m, and with liquid properties close to those of water and gas properties of air, for three different values of liquid viscosity. In Fig. 2c one can indeed see that for $\mathbf{x} < 12.5$ cm, a ten-fold difference in liquid viscosity results in a flow velocity change $\delta < 0.1\%$. The above example also illustrates how eq. 16 requires impractically long capillary length l . To design more compact devices with the same flow properties, the capillary can be truncated at position $x = l_P$, if a shorter and smaller diameter tube with radius $r_R = \alpha \cdot r$ and length $l_R = (l - l_P) \cdot \alpha^4$ is concatenated. Replacing the long downstream capillary section with such a short, narrow vent does not change the viscous losses in the system. Because in most cases $l_P \ll l$, one can choose $\alpha \approx (\frac{l_R}{l})^{1/4}$ as an appropriate value. For the example in Fig. 2c-d, choosing $\alpha = 0.045$ reduces the total capillary length from 6600 m to 15 cm. For the flow in the section $x < l_P$, the governing equations 1 and 2 remain unaltered by such truncation, except for a correction factor $\beta = \alpha^2 + (1 - \alpha^2) \cdot l_P^* < 1$ in the

inertial term. Indeed, Eq. 1 is adapted as

$$P_c = \frac{d}{dt}[(\rho_L \cdot \mathbf{x} + \rho_G \cdot (l_P - \mathbf{x}) + \rho_G \cdot l_R \cdot \frac{1}{\alpha^2}) \cdot \frac{d\mathbf{x}}{dt}] + g \cdot (\mu_L \cdot \mathbf{x} + \mu_G \cdot (l_P - \mathbf{x}) + \mu_G \cdot l_R \cdot \frac{1}{\alpha^2}) \cdot \frac{d\mathbf{x}}{dt} \quad (17)$$

where the factor $1/\alpha^2$ is introduced to account for the increase in gas velocity in the small diameter tube. After integration, eq. 17 can be rewritten as

$$\frac{P_c}{l^2} = \frac{d}{dt}[\rho \cdot (\mathbf{x}^* + \beta \cdot \rho^*) \cdot \frac{d\mathbf{x}^*}{dt}] + g \cdot \mu \cdot (\mathbf{x}^* + \mu^*) \cdot \frac{d\mathbf{x}^*}{dt}, \quad (18)$$

which is identical to eq. 2 except for the factor $\beta < 1$ that reduces the inertial term. Since our novel pump design only operates in the viscosity dominated regime, the pump behaves identically with or without such truncation. This establishes a novel, compact, viscosity independent capillary pump system. Fig. 2d illustrates that the flow regime in the entire region $x \in [r \quad l_P]$ is gas viscosity dominated when adding such downstream air vent channel.

Equations 15 and 16 define a maximum value r_{max} for r and a minimum value l_{min} for l , respectively, for which design the flow velocity, $u_{visc} \approx k_{visc}$, reaches a maximum

$$u_{max} = \frac{\gamma \cdot \cos(\theta)}{4 \cdot \mu_G} \cdot \frac{r_{max}}{l_{min}^2} = \frac{2 \cdot \sqrt{\gamma \cdot \cos(\theta)} \cdot \delta^2 \cdot \mu_G^2}{l_p^2 \cdot \mu^2 \cdot \sqrt{\rho_G}}, \quad (19)$$

with related maximum volumetric flow rate

$$Q_{max} = u_{max} \cdot \pi r_{max}^2. \quad (20)$$

Herein, l_{min} can be derived from the minimum value for l_P , $l_{P,min}$, which in turn can be estimated from the required liquid volume to be pumped, V :

$$l_{P,min} = \frac{V}{\pi r_{max}^2}. \quad (21)$$

For typical values encountered in diagnostic lateral flow applications, e.g. $\delta = 5\%$, liquid density and viscosity close to those of water, a pump volume of 50 μL , and a typical value $\gamma \cdot \cos(\theta) = 30 \text{ mJ/m}^2$, one can calculate $r_{max} = 0.75 \text{ mm}$, and the minimum value for l_P , $l_{P,min}$, can be estimated as $l_{P,min} = \frac{V}{\pi r_{max}^2} = 28.2 \text{ mm}$. We find $u_{max} = 0.33 \text{ mm/s}$ and $Q_{max} = 35.1 \mu\text{L/min}$, i.e. with a pump filling time of 85.4 s, which values are similar to those found in many lateral flow diagnostic applications.

In practical capillary-driven microfluidic devices, the capillary pump performance may be subjected to additional constraints. For example, diagnostic applications may require small upstream channel dimensions to ensure sufficient mass transport in the analyte capture region, which may result in a reduced volumetric flow rate.

Whereas the above analysis is performed for capillary geometries with circular cross-section, the results can be readily extended to rectangular cross-sections typically encountered in microfluidic devices. Furthermore, meandering the capillary channel in such devices allows to further reduce the device size.

Experimental

We experimentally studied the liquid viscosity independent capillary pump design by using a series of liquid samples of similar surface energy and contact angle, but of varying viscosity, and measuring the liquid front position at different time intervals during capillary imbibition of those pumps. The experiments were conducted on the novel capillary pump design illustrated in Fig. 1b and theoretically described in Fig. 2b-c. As reference, traditional capillaries were also tested (illustrated in Fig. 1a). The novel capillary pump design were realized by gluing narrow polymer tubes of 63.5 μm inner diameter and 28 mm length (IDEX Health & Science, USA) at the end of glass capillaries of 1.399 mm inner diameter and 125 mm length (Sigma-Aldrich, USA), which were first cleaned by oxygen plasma treatment to decrease variation in contact angle between the different devices. Seven liquid samples of

different viscosity where prepared using water (DI water, 18 M Ω cm, Millipore), 0.05% Triton X-100 (Sigma-Aldrich, USA) and glycerol (ACS reagent, $\geq 99.5\%$, Sigma-Aldrich, USA) in mass percentages from 0%, to 60%. Whereas the surface energy times cosine contact angle product varies with $< 8\%$, the viscosity of the liquid samples varies with $> 1000\%$ (see table 1; see Supporting Information for details on the how these values were obtained).

Table 1 Viscosity and surface energy of the liquid samples.

Mass % of glycerol in liquid	0	10	20	30	40	50	60
Viscosity (mPa·s) ¹⁴	1.005	1.31	1.76	2.5	3.72	6	10.8
Surface energy (mJ/m ⁻²) $\pm 2.4\%$	31.1	33.1	31.9	31.2	33.1	33.1	29.3
Cosine of the contact angle $\pm 7.6\%$	0.953	0.948	0.951	0.965	0.964	0.962	0.964

The capillary filling process was initiated by bringing the devices in horizontal position into contact with a liquid reservoir of constant height (2 mm), schematically illustrated in fig. 3a. Capillary filling over a length of 100 mm was recorded with a digital camera (Canon EOS 600D, Japan) and analyzed to determine the liquid front position versus time and the average volumetric flow rate. For each of the seven liquid mixtures, the experiment was repeated three times.

Results and Discussion

Fig. 3b and c confirm that our pump provides a flow that is constant in time and independent of the liquid viscosity.

Fig. 3b plots the theoretical (eq. 10) and experimentally obtained liquid front positions x versus time for the different liquid samples. The values used in the theoretical model are based on the viscosity and on the measured surface energy and contact angle of the respective sample liquids, where the measurement error of the surface energy and contact angle measurements results in an estimated standard deviation of 8% on the theoretical values. The average volumetric flow rate over all experiments is 0.15 $\mu\text{L/s}$. For experiments

Figure 3 Experimental results. a) The experimental setup for liquid imbibition with constant upstream liquid pressure conditions close to atmospheric pressure. b) Theoretical and experimental liquid front position versus time for liquid samples with largely varying viscosity. c) Average experimental capillary filling time $t_{x:0 \rightarrow 100\text{mm}}$ from measurements shown in plot b and for normal capillaries as reference (without air vent), normalized against the average filling time of the 0% glycerol mixture, plotted against the viscosity of the sample. Inset d) is a zoom in on the measurements shown in plot c.

using the same liquid, the standard deviation varies between 0.5 and 4.2%. This variations can be ascribed to variations in surface properties of the inner surface of the capillaries. For all experimental values, the standard deviation is 10.8%. This variation can additionally be ascribed to differences in surface energy and contact angle between the liquids. In our experiments, we oxygen plasma treated the capillary surfaces to reduce differences in surface energy, and we added Triton-X 100 to the liquid samples to reduce differences in liquid-solid contact angle between our experiments. This is necessary to demonstrate specifically the viscosity independence of our device. In future applications of the technology, whereas variations in viscosity will not influence the pump performance, natural variations in surface energy and contact angle of unknown sample liquids will remain a source of variation in performance.

Fig. 3c plots the average volumetric flow rate, normalised against the average volumetric flow rate for sample with 0% glycerol, against the liquid viscosity, in which the error bars indicate the standard deviation within the triplicate measurements for every liquid sample. Despite a $> 1000\%$ difference in liquid viscosity, the average volumetric flow rate of the different liquids varies $< 5\%$.

Summary and Conclusions

In summary, we introduce a novel compact viscosity independent constant flow capillary pump design, and validate its behavior both theoretically and experimentally. The experimental results are consistent with the theoretical prediction. This capillary system design can be applied in several engineering fields where a constant liquid flow rate is necessary and we expect our results to find most immediate applications within lab-on-a-chip systems and diagnostic devices.

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Supporting Information Available

Experimental procedures and calculation of surface energy and viscosity of the liquid samples.

This material is available free of charge via the Internet at <http://pubs.acs.org/>.

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