Electronic Supplementary Information (ESI)

Low Yield Stress Measurements with a Microfluidic Rheometer

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This Electronic Supplementary Information (ESI) includes the supplemntary text (Section 1 to 11), figures (Fig.S1 to S14), table (Table 1) , and movies (M1 and M2) for supporting the main manuscript.

1. Yielding behaviour in planar vs. Hele-Shaw linear extensional flow

It is instructive to elucidate the contrast between 2-D or planar extensional flow, which the cross-slot extensional rheometer (CSER) and Optimised Shape Cross-slot Extensional Rheometer (OSCER) produce at the centre, and Hele-Shaw extensional flow, which can be approximated for our microfluidic extensional flow device (MEFD). Let us consider the Bingham plastic model, where the material is Newtonian above a stress τ_y with a viscosity μ, and exhibits zero shear rate at $\tau < \tau_y$. Bingham plastic model in three-dimensional shear[1] is given as

$$\dot{\gamma}_{ij} = 0 \text{ if } \tau < \tau_y, \text{and } \tau_{ij} = \tau_y \frac{\dot{\gamma}_{ij}}{\dot{\gamma}} + \mu\dot{\gamma}_{ij} \text{ if } \tau > \tau_y. \quad \text{(S1)}$$

Here, τ_{ij} and $\dot{\gamma}_{ij}$ are deviatoric stress tensor and strain tensor, respectively. The magnitude of the strain rate, $\dot{\gamma}$, and shear stress, τ, are given as

$$\dot{\gamma} \equiv \sqrt{\sum_{i,j} \frac{1}{2}\dot{\gamma}_{ij}^2}, \quad \text{(S2)}$$

$$\text{and } \tau \equiv \sqrt{\sum_{i,j} \frac{1}{2}\tau_{ij}^2}. \quad \text{(S3)}$$

An ideal unbounded planar extensional flow is given as

$$u_X = -GX, \qquad u_Y = GY. \quad \text{(S4)}$$

As $\dot{\gamma}_{ij} = \frac{1}{2}(\nabla_i u_j + \nabla_j u_i)$, $\dot{\gamma}_{ij}$ is given as

$$\dot{\gamma}_{ij} = \begin{bmatrix} -G & 0 & 0 \\ 0 & G & 0 \\ 0 & 0 & 0 \end{bmatrix}, \quad \text{(S5)}$$

where G is a constant extensional rate. We can evaluate $\dot{\gamma}$ using Eqn. S2. This comes out to be equal to G, which is constant and invariant of the location (X, Y). Thus, τ_{ij} is given as

$$\tau_{ij} = \begin{bmatrix} -(\tau_y + \mu G) & 0 & 0 \\ 0 & (\tau_y + \mu G) & 0 \\ 0 & 0 & 0 \end{bmatrix} \quad \text{(S6)}$$

We know that the rate of viscous dissipation per unit volume, ϕ, is given as

$$\phi = \tau_{ij} : \nabla_i u_j, \quad \text{(S7)}$$

and

$$\tau_{ij} : \nabla_i u_j = 2\mu G^2 + 2G\tau_y. \quad \text{(S8)}$$

Therefore, ϕ is given as

$$\phi = 2\frac{\tau_y^2}{\mu}\frac{(1 + Bn)}{Bn^2}, \quad \text{(S9)}$$

where Bn is Bingham number, and it is defined as

$$Bn = \frac{\tau_y}{\mu G}. \quad \text{(S10)}$$

For yielding, ϕ has to be greater than some characteristic ϕ_y close to yielding. But it is noteworthy that ϕ is not a function of position (X, Y), and it only depends on the values of Bn and material properties τ_y and μ. Thus, this is likely be an on-off flow field, where the material will either yield everywhere or unyield everywhere. This is different from a Hele-Shaw extensional flow as it will be clear soon.

In contrast to the CSER and OSCER, the aspect ratio $\alpha = d/W \ll 1$ for the MEFD (d and W are depth and width of the square channel in the MEFD), which enables a Hele-Shaw approximation of the linear extensional flow such that X and Y directional velocity components (see Fig.S1A for the coordinate system) are as follows:

$$u_X = -GX\left(1 - \left(\frac{2Z}{d}\right)^2\right), u_Y = GY\left(1 - \left(\frac{2Z}{d}\right)^2\right). \quad \text{(S11)}$$

These velocity profiles are assuming a Newtonian flow, but they will still be useful for at least the order of magnitude estimations. $\dot{\gamma}_{ij}$ is given as

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$$\dot{\gamma}_{ij} = \begin{bmatrix} -G\left(1-\frac{4Z^2}{d^2}\right) & 0 & \frac{4G}{d^2}XZ \\ 0 & G\left(1-\frac{4Z^2}{d^2}\right) & \frac{-4G}{d^2}YZ \\ \frac{4G}{d^2}XZ & \frac{-4G}{d^2}YZ & 0 \end{bmatrix} \quad \text{(S12)}$$

In this case, $\dot{\gamma}$ is given by $G\xi$, where $\xi = \sqrt{\left[1-\frac{4Z^2}{d^2}\right]^2 + \frac{16}{d^4}Z^2(X^2+Y^2)}$. Thus, $\dot{\gamma}$ is a function of X, Y, and Z, unlike the previous case of G for unbounded planar extensional flow. Similarly, τ_{ij} is given as

$$\tau_{ij} = \begin{bmatrix} \tau_{11} & \tau_{12} & \tau_{13} \\ \tau_{21} & \tau_{22} & \tau_{23} \\ \tau_{31} & \tau_{32} & \tau_{33} \end{bmatrix} \quad \text{(S13)}$$

where $\tau_{11} = -\frac{\tau_y\left(1-\frac{4Z^2}{d^2}\right)}{\xi} - \mu G\left(1-\frac{4Z^2}{d^2}\right)$, $\tau_{12} = \tau_{21} = \tau_{33} = 0$, $\tau_{22} = \frac{\tau_y\left(1-\frac{4Z^2}{d^2}\right)}{\xi} + \mu G\left(1-\frac{4Z^2}{d^2}\right)$, $\tau_{13} = \tau_{31} = \frac{\tau_y\frac{4}{d^2}XZ}{\xi} + \mu\frac{4G}{d^2}XZ$, and $\tau_{32} = \tau_{23} = -\frac{\tau_y\frac{4}{d^2}YZ}{\xi} - \mu\frac{4G}{d^2}YZ$. Like we discussed before, we can find ϕ for unbounded Hele-Shaw extensional flow. It is given as

$$\phi = 2\frac{\tau_y^2}{\mu}\frac{(Bn\xi + \xi^2)}{Bn^2}. \quad \text{(S14)}$$

Here, ϕ is a function of the position (X, Y, Z), where $X, Y \in (-\infty, +\infty)$ and $Z \in [-d/2, +d/2]$. When $Z = 0$, we get $\xi = 1$, and ϕ for the Hele-Shaw case converts to Eqn. S9, i.e., the planar extensional flow. Again, in this case, we can assume that there is a characteristic ϕ_y depicting yielding such that there exists a region for which $\phi < \phi_y$ exhibiting solid-like behaviour, and $\phi > \phi_y$ showing a liquid-like behaviour.

To assign some approximate value of ϕ_y for order of magnitude estimates, we can use only the first part of the tensorial Bingham plastic model in Eqn. S7, which leads to

$$\phi_y = \tau_y\frac{\dot{\gamma}_{ij}}{\dot{\gamma}}:\nabla_i u_j. \quad \text{(S15)}$$

Assuming $\xi \sim O(1)$ closer to the center up to a distance d, ϕ_y is given as

$$\phi_y \approx 2\frac{\tau_y^2}{\mu Bn}. \quad \text{(S16)}$$

Hence, any region for which $\phi < 2\frac{\tau_y^2}{\mu Bn}$ is the unyielded region. We can compare this analytical solution with the experimental results. For example, 0.3% carbopol exhibited $\tau_y \approx 1.29$ Pa and $\mu \approx m \approx 2.49$ Pa − s (using the Herschel-Bulkley parameters). Further, the extensional rate for $\Delta P = 241$ kPa is $G \approx \frac{U_{at\,M}}{D_{M-O}} \approx 0.055\ \mathrm{s^{-1}}$. Thus, $Bn \approx 10$. Fig.S1B shows the contours of ϕ plotted on $X - Y$ plane. ϕ increases (see the colourbar in the figure) as we go away from the center (0,0). In Fig.S1C, we plot ϕ, for the same experimental case, on $X - Z$ plane at $Y = 0$. It turns out that the unyielded region is smaller at the top plane or $Z = d/2$, and grows in size as we move towards the midplane, and diverges at $Z = 0$, as Hele-Shaw flow becomes planar extensional flow at $Z = 0$. In our experiments, we captured the flow field approximately at the midplane. Therefore, for comparison with the theory, we want to pick a Z value 'close enough' to the centre, but not exactly at the centre. Thus, we arbitrarily pick $Z = d/20$ for Fig.S1B. We also show ϕ_y by a dotted circle. Any region within this circle should exhibit unyielding, and the region exterior to circle should be flowing. We can compare this region with the experimental unyielded region as shown by the square, and they appear to be of the same order in size. We performed similar comparisons between experimental and theoretical S, where we measured a scale of G using $\frac{U_{at\,M}}{D_{M-O}}$ for a range of ΔP for 0.3% CS. We provide this comparison in Fig.S1D, which shows reasonable agreement between the experiment and theory. However, it is important to reiterate that these calculations serve as order of magnitude estimates to assess the unyielding in the unbounded Hele-Shaw flow, and comparing the unyielded region size with the experimental observation. In practice, the velocity profiles will be different from Eqn. S11, and the calculation of ϕ_y will require further refinement.

2. Yielding behaviour in the MEFD

We performed a simulation in COMSOL to verify our experimental observation that the material unyields at the centre. In Fig.S2A, we demonstrate the unyielded and yielded regions at $\Delta P/\tau_y W d^2 \mathcal{R}_o = 0.015$. We find that there exists a stagnant, non-moving, and unyielded region forming at the stagnation point, as seen also in the experiments (see Fig.1E-G). We demarcate these regions in the figure using the definition of yielded zone in COMSOL, which is different from our definition of unyielding based on a strain rate criterion. Thus, the size of the unyielded region is likely be different numerically. But we can still get some qualitative information. For example, in the figure, we also observe another four unyielded regions at the four walls or sides of the square channels. Let us term them as 'pseudo' regions, as they are not visible in an experiment. These regions are the result of no-slip boundary condition at the wall, which means, somewhere between the wall and the stagnation point, there must be a velocity maximum (which we observe to be at M and N in the experiment and simulation, see Fig.2C). This means that the change in the velocity is nearly zero, and the shear stress will be below the yield stress. But these regions do not really move. The fluid enters these regions, begins to move like a plug of a yield stress fluid flowing through a circular conduit, and then comes out again as yielded. This is similar to the discussion on Fig.1 of Denn and Bonn (2010).[2] These regions cannot be predicted by Eqn. S14 since they are a consequence of four sides or walls of the channel bounding the flow. The unyielded region at the centre is stagnant and non-moving, and thus can be visualized easily using a visualization technique. Hence, we use it in the measurement of the yield stress. In Fig.S2B, we present the shear stress, τ_{A-B}, plotted vs. D_{A-B}

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(shown along the dotted line $A-B$ in Fig.S2A). The yield stress line is shown for the reference (for 0.3% CS). As discussed in Section 2 of the main text, τ_{A-B} is higher at the wall or point A, and reduces as we move away from the wall. It drops below the yield stress line, and is minimum where U is maximum. This is a pseudo region that exists near the side of the square. Further, τ_{A-B} increases and becomes maximum, and further drops below the yield stress as we approach the stagnation point. This is the stagnant and unyielded region that exists at the centre.

3. Demarcating the stagnation region

In our experiments, we used a strain rate criterion to measure the size of the stagnant region, where we approximate the strain rate with a scale. This scale is easy and convenient to calculate, as compared to the complicated definition of $\dot{\gamma}$ given in Eqn. S2. We can show using the scaling analysis that this approximation is reasonable. In Hele-Shaw extensional flow in the MEFD, velocity u_X and u_Y scale as U, X and Y scale as W, and Z scales as d, which also means

$$\frac{\partial u_X}{\partial X}\sim\frac{U}{W},\frac{\partial u_Y}{\partial Y}\sim\frac{U}{W},\frac{\partial u_X}{\partial Z}\sim\frac{U}{d},\text{and }\frac{\partial u_Y}{\partial Z}\sim\frac{U}{d}. \quad \text{(S17)}$$

Since $W \gg d$, $U/W \ll U/d$, and hence, $\partial u_X/\partial Z$ and $\partial u_Y/\partial Z$ have a major contribution towards $\dot{\gamma}$. Therefore, $\dot{\gamma}$ will scale as

$$\dot{\gamma}\sim\frac{U}{d}, \quad \text{(S18)}$$

for a large portion of the device, except in regions close to the stagnation point and close to the wall. This could be confirmed by the simulation as shown in Fig.S2C. We show both $\dot{\gamma}$ calculated using the definition given in Eqn.S2 and S18. They appear to be close and of the same order of magnitude in the region, mainly where they intersect $\dot{\gamma}_{CL}=10^{-1}\mathrm{s}^{-1}$. Thus, the measurement of the size of the unyielded region should be fairly accurate with this simple shear rate scale. Furthermore, in this scale, we make an assumption that the velocity profile is parabolic along the Z-axis in the square channel, where $\tau_w > \tau_y$, at least at the boundary of the stagnant region, where τ_w is the wall stress at top or bottom plane. But it is likely that $\tau_w\sim\tau_y$, leading to a velocity profile with a plug in the middle, and quadratic velocity profile elsewhere (a discussion can be found out in ESI Section 9 or Fig.S12 pertaining to a similar plug flow in a tube). In such a case, the strain rate scale will change to $U/[d(1/\psi-1)^2]$, where $\psi=\tau_y/\tau_w$.[3]

4. Scaling relationship

In this section, we employ scaling analyses using a Hele-Shaw extensional flow approximation (aspect ratio $\alpha \ll 1$). To begin, we can first calculate an Re for the flow of water through the MEFD as

$$Re=\frac{\rho_W U d}{\mu_W}\sim 10^{-5}\text{ to }10^{-1}\ll 1, \quad \text{(S19)}$$

where ρ_W is the density of water ($\sim10^3\mathrm{kg/m^3}$), U is the magnitude of the velocity through the MEFD ($\sim10^{-1}$ to 10^3 μm/s), d is the depth of the MEFD ($\sim10^2$ μm), and μ_W is the viscosity of water ($\sim10^{-3}$ Pa − s). Thus, we can assume a creeping flow regime. Further, in a typical experiment, ΔP was reduced and the unyielded region grew from a small size ($S/W \ll 1$) to a large size comparable to the width of the square ($S/W\sim1$) as shown in Fig.4C. For the simplicity of calculations, let us assume a Bingham plastic model such that when $\tau>\tau_y$, μ is finite and constant. In the limits of $S/W \ll 1$, we assume a linear extensional flow around the unyielded region in the square channel of the MEFD (see Fig.4A). τ at the center of the square channel (τ_c) is the extensional stress along X and Y and it scales as

$$\tau_C\sim\mu G, \quad \text{(S20)}$$

where G is the constant extensional rate in the MEFD, and μ is the viscosity of the fluid. As we move away from the center, the shear stress in depth direction or Z starts to dominate. Assuming a stagnant region for which $S \ll W$, but $S > d$, appears, the shear stress at the boundary of the stagnant region (τ_b) is given as

$$\tau_b\sim\frac{\mu G S}{d}. \quad \text{(S21)}$$

G is related to U, and the relevant length scale for the extensional direction is W. Thus, G is given as

$$G\sim\frac{U}{W}. \quad \text{(S22)}$$

U scales as Q/A_c, where Q is the flow rate and A_c is the area of cross section available for flow. A_c in the MEFD scales as Wd, and thus, U is given as

$$U\sim\frac{Q}{Wd}. \quad \text{(S23)}$$

The next step is to relate Q with the experimental parameter, ΔP. Under the creeping flow, Q varies linearly with ΔP, and is given as

$$Q=\frac{\Delta P}{\mu\mathcal{R}_o}, \quad \text{(S24)}$$

where $\mathcal{R}_o$ is the resistance (in the units of $\mathrm{m^{-3}}$) to the fluid flow. In our experimental setup, there are multiple resistances in series. The types of resistances are described below.

a) PEEK tubing (see Fig.1C) of a circular cross section of dimensions, L_t and d_t. The resistance for PEEK tubing is $\mathcal{R}_{Tube}=128L_t/\pi d_t^4$ as given by Hagen–Poiseuille equation for Newtonian fluids.

b) Side channel with a square cross-section (inlet or outlet channel) of dimensions L, b, b ($b\approx d$). For the side channel, we assume $\mathcal{R}_{Side}=L/(\lambda_{side}b^4)$, where $\lambda_{side}=0.035144$.

C) Square shape of the MEFD with a rectangular cross-section of dimensions W, W, d. Assuming flow through the MEFD square channel is similar to a channel with a rectangular cross-section of area Wd and length W, we assume $\mathcal{R}_{Square}=$

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$12/(1-\lambda_{sq}d/W)d^3$, where $\lambda_{sq}=0.63$. Since there is an inlet PEEK tubing, an inlet side channel, a square channel, an outlet side channel in series (see Fig.1C), the total resistance in series is

$$\mathcal{R}_o = \mathcal{R}_{Tube} + 2\mathcal{R}_{Side} + \mathcal{R}_{Square}. \quad \text{(S25)}$$

In many experiments presented in this paper, we did not use an outlet PEEK tubing (as shown schematically in Fig.1C) to prevent the fluid from leaking between the microfluidic device and the outlet PEEK tubing. In case we use it, we modify the total resistance to include the outlet $\mathcal{R}_{Tube}$ in series. The total resistance, $\mathcal{R}_o$, is given as

$$\mathcal{R}_o = \frac{128L_t}{\pi d_t^4} + \frac{2L}{\lambda_{side}b^4} + \frac{12}{\left(1-\frac{\lambda_{sq}d}{W}\right)d^3}. \quad \text{(S26)}$$

From Eqn. S24 and S26, Q is given as

$$Q = \frac{\Delta P}{\mu\left(\frac{128L_t}{\pi d_t^4} + \frac{2L}{\lambda_{side}b^4} + \frac{12}{\left(1-\frac{\lambda_{sq}d}{W}\right)d^3}\right)} = \frac{\Delta P}{\mu\mathcal{R}_o}. \quad \text{(S27)}$$

Substituting Q in Eqn. S23 yields

$$U \sim \frac{\Delta P}{\mu\mathcal{R}_o W d}, \quad \text{(S28)}$$

and thus, using U and combining Eqn. S22, we get

$$G \sim \frac{\Delta P}{\mu W^2 d\mathcal{R}_o}. \quad \text{(S29)}$$

From Eqn. S21, we get

$$\tau_b \sim \frac{\Delta P}{W^2 d\mathcal{R}_o}\frac{S}{d}. \quad \text{(S30)}$$

Since we are in the limits where we observe the existence of the unyielded region, the shear stress at the boundary of the unyielded region should be of the same order as the yield stress of the fluid ($\tau_b \sim \tau_y$ or $\tau_b/\tau_y \sim 1$). Thus, Eqn. S30 can also be written as

$$\frac{\Delta P}{\tau_y W^2 d\mathcal{R}_o}\frac{S}{d} \sim 1. \quad \text{(S31)}$$

After rearranging the previous equation, we get

$$\frac{S}{W} \sim \frac{1}{\frac{\Delta P}{\tau_y W d^2 \mathcal{R}_o}}. \quad \text{(S32)}$$

Further, we know from the experiments that when $\Delta P > \Delta P_{crit}$, $S=0$. Therefore, we can write S as a function of ΔP and τ_y with the following relationship up to two unknowns, C_0 and C_1.

$$\frac{S}{W} = -C_0 + \frac{C_1}{\frac{\Delta P}{\tau_y W d^2 \mathcal{R}_o}}. \quad \text{(S33)}$$

5. Calculation of ΔP_{crit}

From Eqn. S33 in ESI Section 4, at $\Delta P = \Delta P_{crit}$, $S/W = 0$, and thus, we get

$$\Delta P_{crit} = \left(\frac{C_1}{C_0} W d^2 \mathcal{R}_o\right)\tau_y. \quad \text{(S34)}$$

Assuming an approximate yield stress value before actually measuring it, we can calculate ΔP_{crit} using Eqn. S34. This is useful during the experiment, where we can begin recording the videos below ΔP_{crit}. For example, for PEEK tubing of $d_t = 127\ \mu\text{m}$,

$$\Delta P_{crit}(\text{kPa}) \approx 5000\ \tau_y(\text{Pa}). \quad \text{(S35)}$$

If we wish to measure τ_y of a material, and there is sufficient literature to suggest that τ_y should be of the order of 0.1 Pa, then we can start with a maximum $\Delta P = 500$ kPa and reduce it further to record the flow.

6. Rheometer limit, slip, and other rheology measurements

We used a standard parallel plate rheometer (Discovery Hybrid Rheometer-3) to conduct rheological assessments. Our observations revealed that we could reliably measure τ_y of carbopol within a range of C_{CS} between 0.3% and 0.015%. However, C_{CS} below 0.015% posed challenges, as we encountered the lower limit of shear stress detection imposed by the rheometer, as depicted in Fig.S3A. Specifically, at 0.01% CS, the measured shear stress coincided with the instrument's limit, rendering these data points unreliable and unsuitable for inclusion in our analyses. Furthermore, rheological responses below 0.01% concentration consistently fell below the instrument's detection limit, plateauing at a stress of the order of 0.005 Pa. Similarly, additional factors such as slip in both standard and MEFD rheometers, as well as operational gaps, can influence the rheology of carbopol. Moreover, other rheological measurements, including linear and non-linear oscillatory studies, are important for our understanding of carbopol's rheology. These discussions are elaborated in the subsequent sections.

6.1 Estimation of rheometer limit

We can estimate the minimum shear stress, denoted as τ_{min}, that our parallel plate rheometer can reliably measure using the minimum torque, T_{min}, specified by the manufacturer, which is 5 nN − m. Using the expression, $\tau_{min} = 2T_{min}/\pi a_{plate}^3$, where a_{plate} represents the radius of the top plate, we calculate τ_{min} to be 0.1 mPa. However, it is important to acknowledge that the actual minimum stress could potentially a few orders higher in magnitude than this theoretical minimum stress, which may influence our measurements of dilute carbopol samples.[4–8] Thus, we determined the actual τ_{min} using four reference fluids: Siltech F-1000 (Siltech), light mineral oil (ACP), white mineral oil (Fisher Scientific), and water (Milli-Q®). These fluids

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were subjected to $\dot{\gamma}$ ranging between $100 - 10^{-7}\mathrm{s}^{-1}$, and the resulting rheological data is presented in Fig.S4A. Notably, we found that τ plateaus at $\tau \sim 1\ \mathrm{mPa}$, an order higher than the lowest limit we calculated from T_{min}. Additionally, solid lines in the figure represent τ predicted using the Newtonian viscosity, denoted as τ_{pred}. Here, we calculated the Newtonian viscosity using the reverse order of the first five data points starting from $\dot{\gamma} = 100\ \mathrm{s}^{-1}$. In Fig.S4B, we illustrate the discrepancy between the measured or actual shear stress (τ_{act}) and τ_{pred}, which is given by $|\tau_{act} - \tau_{pred}|/\tau_{act}$ (in %) vs. τ_{act}. It is evident that the error exceeds 10% when τ_{act} fall between $10^{-3} - 10^{-2}$ Pa for all the four fluids. Hence, we establish 0.005 Pa as a conservative lower limit or τ_{min} (corresponds to $212\ \mathrm{mN-m}$), below which there is considerable uncertainty in τ measurement. In light of these considerations, it is evident from Fig.3A that τ data for 0.015% concentration lies above τ_{min}. However, for the 0.01% concentration, τ coincides with τ_{min}. Moreover, we observed that for concentrations ranging between 0.0015% and 0.005% (data not shown), the shear stress at lower shear rates (below $1\ \mathrm{s}^{-1}$) plateaus between 0.003 and 0.008 Pa (averaging roughly about 0.005 Pa). Consequently, we exclude carbopol solutions with concentrations below 0.015% from our analysis due to the inherent uncertainty in τ measurements at such dilute concentrations.

6.2 Slip and operation gap

When the shearing surface is smooth, it can lead to slipping of the material closer to the surface, affecting the rheological measurements. Previous studies have shown that slip effects can cause apparent rheological behaviour, particularly at low shear rates, often resulting in a noticeable kink in the shear stress profile. However, this kink tends to disappear when rough shearing surfaces are employed, as demonstrated in studies involving soft microgel particles.[9–11] Moreover, reducing the operating gap can exacerbate the slip effect, especially with smooth surfaces.[12] These considerations are important for our rheometer measurements, given our use of a small operating gap of $117\ \mu\mathrm{m}$ and a sandblasted top plate combined with a smooth peltier plate, which may lead to slipping. In Fig.S4C, we address these concerns. We observed a kink in the rheological data for concentrations of 0.025 and 0.05% when using an operating gap of $117\ \mu\mathrm{m}$ and a sandblasted plate-smooth peltier plate assembly. Upon gluing 600 Grit sandpaper to both the top and bottom plates[13], we observed the absence of such a kink. Subsequently, for the remaining concentrations, we did not observe a significant kink. Therefore, we opted to maintain the use of a sandblasted top plate and smooth peltier plate for these measurements. Furthermore, we conducted a comparison of the rheological behaviour of a 0.1% solution with operating gaps of $117\ \mu\mathrm{m}$ and $1\ \mathrm{mm}$, finding relatively consistent results. Hence, we decided to maintain an operating gap of $117\ \mu\mathrm{m}$ for all measurements. This choice allows direct comparison with our MEFD measurements, where the depth was also set at $117\ \mu\mathrm{m}$.

It is also essential to investigate the surface roughness of the internal surfaces within the MEFD, particularly the top (PDMS) and bottom (glass) surfaces of the square channel. We did not study the impact of the degree of slip on our yield stress measurements in this study; it is a topic for future work. However, we can assess whether our microfluidic surfaces were rough or smooth. Duangkanya et al. (2022) demonstrated that plasma treatment with a power of 200 W for approximately 24 s resulted in a surface roughness of $600\ \mathrm{nm}$.[14] We employed plasma treatment twice in our device: first during the bonding of PDMS and glass, and secondly during the coating process with silane (refer to Materials and Methods). Thus, it is likely that we introduced a degree of roughness on the PDMS side as well. For the plasma treatment, we utilized a BD-20 High Frequency Generator (Electro Technip Products) to treat the glass and PDMS surfaces for over a minute during the bonding step, and nearly 30 s during the coating process. Assuming a similar effect to the aforementioned study, we anticipate a surface roughness of the order of $1\ \mu\mathrm{m}$. Consistent with these findings, our observation revealed rough PDMS side in our device, as illustrated by two examples of our used devices in Fig.S4D. The scratches observed in Fig. S4D are present throughout the device but are more noticeable along the diagonals and a smaller square inside, which are generated during the coating step through the four ports. To measure the surface roughness, we conducted additional experiment using a PDMS slab subjected to plasma treatment for one minute and then 30 s to simulate bonding and coating stages. Subsequently, we examined these PDMS slabs under a 5000X lens in the Keyence VHX7000 microscope system. Our analysis revealed that the roughness was of the order of $1\ \mu\mathrm{m}$ in size, consistent with our earlier predictions. Similarly, Alam et al. (2014) demonstrated the introduction of surface roughness on glass of around $10\ \mathrm{nm}$ within 60 s using 300 W plasma activation.[15] This observation suggests that glass surfaces tend to be relatively smoother compared to PDMS. Now, let us compare these roughness values with those of the rheometer surfaces. The surface roughness for Grit 600 sandpaper is nearly $130\ \mathrm{nm}$, whereas for the sandblasted plate, it ranges between $1.4 - 1.8\ \mu\mathrm{m}$. This indicates that the roughness of the MEFD surfaces is comparable to that of the rheometer surfaces. However, we acknowledge the necessity of a separate study to investigate the relationship between slip and yield stress measurements.

6.3 Oscillatory rheology

The literature suggests the presence of a percolating network in carbopol at low concentrations, transitioning to jamming at higher concentrations.[16] This microstructural network, responsible for the yield stress of carbopol, also leads to

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elasticity, with both properties generally exhibiting similar scaling with concentration and pH.[16] Consequently, the existence of a yield stress implies a non-zero shear modulus (G') greater than the loss modulus (G"), which remains relatively constant at lower frequencies of oscillation (ω).[16] Further, both G' and G" are nearly constant at lower strain γ such that $G' >$ G", but they crossover at higher γ ($> 10\%$). We also produce these rheological measurements as discussed by Gutowski et al. (2012).[16] Fig.S5A depicts G' and G" as a function of γ. We observe that G' was roughly an order higher than G" higher at low γ ($< 10\%$) for 0.1 and 0.3% CS, while they were of similar orders of magnitude for 0.05% CS. At higher γ, G' crosses over G" at a crossover strain γ_{cr} (such that $G' = G" = G_{cr}$). We can determine the crossover stress, which essentially represents the yield stress, as demonstrated by Dinkgreve et al. (2016).[17] For instance, for 0.3% CS, the calculated value of τ_y was 1.9 Pa, compared to 1.3 Pa measured using steady shear rheology. Further, in Fig.S5B, we present G' and G" as a function of frequency of oscillation (ω). We can observe that $G' > G$" and both G' and G" are nearly constant at lower ω for $0.05 - 0.3\%$ CS, indicative of gel-like behaviour as observed by Divoux et al. (2011).[18] However, when examining the data for 0.025% CS we could plot only some of the data at higher ω and γ, due to the proximity of the data to the low torque limit close to 212 nN $-$ m that we measured in Section S6.1. But from the available data, it appears that $G' \sim G$", and thus 0.025% CS may be gel-like or liquid-like. Nevertheless, we still consider yield stress of 0.015% and 0.025% CS in our measurements. However, it may be appropriate to limit the discussion of these concentrations to apparent yield stress.

7. Other aspects of real-time measurement of τ_y

In our study, we have successfully demonstrated the measurement of low yield stress. However, the MEFD rheometer offers another significant advantage: the ability to measure the yield stress in real-time as the material flows continuously through our device. In this section, we will delve into some of the aspects of these measurements in further detail.

7.1 Measurement of τ_y vs. number of recorded frames

We used 0.1% CS and directed it to flow through the MEFD at a range of ΔP, and recorded a long video of a few minutes using a Nikon microscope with FITC filter and a Lumenera camera (Infinity Analyze 7). Subsequently, we analysed these videos by measuring velocities and averaging them across different numbers of images, ranging from 2 to 2000. The measured values of S, obtained for different numbers of images recorded at a range of ΔP, are presented in Fig.S6A. We observed that the magnitude of S, measured for a range of images between $2 - 2000$, were reasonably close. Furthermore, we derived estimates of the yield stress from this data, as illustrated in Fig.S6B. It appears that τ_y by averaging velocities over 2 images closely approximated that measured using 2000 images. Additionally, for comparison purposes, we included the yield stress measured using the rheometer, and we observed a good agreement between the two, as indicated by the overlapping error bars.

7.2 Ramp up and ramp down in ΔP

Another important aspect to consider is the measurement of thixotropy in yield stress fluids and investigation of rheology based on the shear history.[19] Bonn et al. (2009) investigated thixotropy in carbopol gel by conducting ramp up and ramp down sweeps of shear stress. They observed that both curves overlapped, ruling out thixotropy for carbopol gel.[20] In all our experiments presented in the main manuscript, we begin the material flow at a significantly high ΔP, and gradually reduce it step by step while recording videos at each ΔP. Essentially, our measurements represent a ramp down of shear stress. However, to further explore this phenomenon, we also examined ramp up and ramp down shear stress for two CS with concentrations of 0.3 and 0.05%. We measured τ_y in real-time averaging the data over 100 images. For the ramp up scenario (see Fig.S7A), we increased ΔP from 69 kPa to 207 kPa for 0.3% CS and from 14 kPa to 34 kPa for 0.05% CS. Upon immediately commencing video recording after increasing ΔP, we observed a slight kink at the beginning where τ_y is initially higher but later stabilized to a steady-state value. Similarly, in the ramp down scenario (see Fig.S7B), we decreased ΔP from 517 kPa to 207 kPa for 0.3% CS and from 276 kPa to 34 kPa for 0.05% CS. In this case, we observed a similar kink but in the opposite direction. For comparison, we maintained the same steady state ΔP for both ramp up and ramp down cases. We observed that steady state τ_y is nearly constant for both the concentrations, suggesting no discernible difference between the ramp up and ramp down studies. This reinforces the findings of Bonn et al. (2009). Additionally, the kink observed in the beginning diminishes after a few tens of seconds. As discussed in our Materials and methods (Section 2.5), we implemented a one-minute waiting period after changing ΔP in our experiments to ensure that a steady state was reached.

8. Measurement of τ_y using a wider tube ($d_t = 508$ μm)

For our experiments, we had a certain fixed range of ΔP for our microfluidic flow controller ($0 - 550$ kPa). We used a narrow tube to measure lower yield stress between $10^{-2} - 1$ Pa. We also wanted to test a wider tube of $d_t = 508$ μm, which could potentially be used to measure higher yield stresses (typically above 0.1 Pa). We see in Fig.5A and B that the yield stresses measured using the 508 μm tube are fairly precise and match

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with the rheometer yield stress. In Fig.S10, we show the experimental results associated with it, including the $S-\Delta P$ data, model fit using 0.3% CS, and S/W vs. $\Delta P/\tau_y W d^2 \mathcal{R}_o$ for $0.05-0.3\%$ CS, where the model fit coefficients for $d_t = 508$ μm were $C_0 = -0.133$ and $C_1 = 0.078$. We identified that the model fit associated with the wider tube was different from the model fit of the narrow tube, which should not be the case if we have performed the correct scaling. To understand the reason behind this, we speculated that there could be unyielding in the wider tubes, thereby changing the resistance $\mathcal{R}_o$, which was calculated assuming a Newtonian fluid. We further investigate this effect of unyielding in a circular tube in greater detail in ESI Section 9.

9. Unyielding and increase in the flow resistance in the tubes

In Fig.S12A and B, we show a schematic of the flow of Newtonian and Bingham fluid through a circular tube. Let us say that the radius of the tube is a_o, and the velocity along z is v_z. In the Newtonian case, the velocity profile is parabolic. In the case of Bingham fluids, the material will flow if the wall stress, $\tau_w > \tau_y$. Depending on the ratio $\psi = \frac{\tau_y}{\tau_w}$, we may see the formation of an unyielded plug of radius a_p (such that $\psi = \frac{a_p}{a_o}$), which moves at the constant velocity, v_p. In the yielded region, the velocity profile is parabolic and is given by v_z. In the cylindrical coordinates, the Bingham plastic model for the tube flow is given as

$$\boldsymbol{\tau_{az} = \tau_y + \mu \frac{dv_z}{da}}, \quad \text{(S36)}$$

where τ_y and μ are Bingham yield stress and viscosity. The governing equation to find the modified Hagen–Poiseuille equation for the Bingham flow through the tube is given as

$$\boldsymbol{\tau_{az} = \frac{\Delta P}{2L_t} a}, \quad \text{(S37)}$$

where ΔP is the pressure drop applied across the tube, and L_t is the length of the tube. Thus, we need to solve

$$\boldsymbol{\frac{\Delta P}{2L_t} a = \tau_y + \mu \frac{dv_z}{da}}, \quad \text{(S38)}$$

using the boundary conditions

$$\boldsymbol{\tau_{az}|_{a=a_o} = \tau_w,\ v_z|_{a=a_o} = 0,\ v_z|_{a=a_p} = v_p}. \quad \text{(S39)}$$

Therefore, the flow rate, Q, is given as

$$\boldsymbol{Q = \frac{\pi a_o^4 \Delta P}{8\mu L_t}\left[1 - \frac{4}{3}\psi + \frac{1}{3}\psi^4\right]}, \quad \text{(S40)}$$

where the term $\frac{\pi a_o^4 \Delta P}{8\mu L}$ is of the Newtonian flow. Thus, we define the new Bingham flow resistance in the PEEK tubing as

$$\boldsymbol{\mathcal{R}_{Tube_{BP}} = \frac{\Delta P_T}{\mu Q} = \frac{128 L_t}{\pi d_t^4\left[1 - \frac{4}{3}\psi + \frac{1}{3}\psi^4\right]}} \quad \text{(S41)}$$

$$\boldsymbol{= \mathcal{R}_{Tube_N}\left[1 - \frac{4}{3}\psi + \frac{1}{3}\psi^4\right]^{-1}},$$

where $\mathcal{R}_{Tube_N}$ is the resistance in the case of Newtonian flow. We need to modify our total resistance $\mathcal{R}_o$ according to this new tube resistance, such as

$$\boldsymbol{\mathcal{R}_{o_{BP}} = \mathcal{R}_{Tube_{BP}} + 2\mathcal{R}_{Side} + \mathcal{R}_{Square}}. \quad \text{(S42)}$$

Thus, the dimensionless shear stress will appear as $\Delta P/\tau_y W d^2 \mathcal{R}_{o_{BP}}$. In Fig.S12C, we show the dimensionless data using both $\mathcal{R}_o$ and $\mathcal{R}_{o_{BP}}$ for both the tubes, $d_t = 127$ and 508 μm. We denote the scaled data using $\mathcal{R}_{o_{BP}}$ in the figure by corrected resistance or CR. We also present the model fit for both the tubes. We can infer from the figure that the correction term is not significantly different for the 127 μm, meaning, there is not a significant plug formation in the 127 μm tube. The corrected scaled data lies within the confidence interval of the model fit. Thus, we do not change the model fit for the calculation of the yield stress, and stick with the Newtonian assumption. However, for 508 μm, we see a significant change in the scaled data. The corrected data is closer to the model fit of 127 μm, almost within its confidence interval. This shows that the difference between the model fits could be due to the increased flow resistance and unyielded plug formation in the wider tube. Notably, as discussed in ESI Section 8, when a separate characterization step was followed, it still produced the correct yield stress values for the 508 μm tube (see Fig.5A). But if we want to stick to one model fit as calculated using the 127 μm tube, we must check if there is a significant change in the flow resistance due to the yield stress in the tube size that we might be using. For a tube of any dimensions, we can work out a criterion that needs to be followed to ensure that unyielding is not altering $\mathcal{R}_o$. This criterion is that the wall stress $\tau_W = \frac{\Delta P d_t}{4L_t} \gg \tau_y$. From Eqn. S33 or 5, we can also write this criterion as $\frac{4L_t\left(\frac{S}{W}+C_0\right)}{C_1 W d^2 \mathcal{R}_o d_t} \ll 1$. For $d_t = 127$ μm and $L_t = 1$ m and $\frac{S}{W}$ varying between $0.1-0.7$, $\frac{4L_t\left(\frac{S}{W}+C_0\right)}{C_1 W d^2 \mathcal{R}_o d_t} = 0.045 - 0.27$, which is satisfying the criterion. When L_t is increased, even $\mathcal{R}_o$ increases, and thus, this ratio still remains much smaller than 1. Thus, changing the length of the smaller tube does not lead to any unyielding in the tube or changes to the model fit.

10. Error analysis and uncertainty in the measurement of τ_y

As we discussed in Section 3.5 in main text, for each pair of $S-\Delta P$, we measured one value of τ_y, and thus, many such τ_y were averaged to determine τ_{yM}. We calculated the standard deviation, $S_{\tau_{yM}}$, and percentage error, $S_{\tau_{yM}}/\tau_{yM}$ (%), for different τ_{yM} as shown in Fig.5B. We saw a general trend of increasing $S_{\tau_{yM}}/\tau_{yM}$ from 1 to nearly 50% for decreasing τ_{yM} from $\sim O(1)$ to $O(10^{-2})$ Pa. In our experiments, we used two

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sizes of PEEK tubings. For $\tau_{yM} \sim O(10^{-1})$ to $O(1)$ Pa, we used PEEK tubing of $d_t = 508$ μm, and for $\tau_{yM} \sim O(10^{-2})$ to $O(1)$, $d_t = 127$ μm, and their respective errors are shown by diamonds and circles. We also report the error in the measurement of the yield stress of blood in Fig.5B. For comparison, we calculated $S_{\tau_{yR}}/\tau_{yR}$ (%) using the rheometer for three CS samples (see Fig.5B). The measurement errors for both the rheometer and MEFD were similar in magnitude.

The source of the uncertainty in the measurement of τ_y could be the uncertainty in ΔP, $d(\Delta P)$, and S, dS. The linear propagation of errors gives us the result,

$$\boldsymbol{d\tau_y} = \sqrt{\left(\frac{\partial \tau_y}{\partial(\Delta P)} d(\Delta P)\right)^2 + \left(\frac{\partial \tau_y}{\partial S} dS\right)^2}. \quad \text{(S43)}$$

Using Eqn. 5, we can write $d\tau_y$ in terms of the experimental parameter ΔP and τ_y as

$$\boldsymbol{d\tau_y} = \sqrt{\left(\frac{\tau_y}{\Delta P} d(\Delta P)\right)^2 + \left(\frac{\Delta P}{C_1 W^2 d^2 \mathcal{R}_0} dS\right)^2}. \quad \text{(S44)}$$

Thus, the relative uncertainty is given as

$$\textbf{Uncertainity } (\%) = \frac{\sqrt{\left(\frac{\tau_y}{\Delta P} d(\Delta P)\right)^2 + \left(\frac{\Delta P}{C_1 W^2 d^2 \mathcal{R}_0} dS\right)^2}}{\tau_y} \textbf{X 100}. \quad \text{(S45)}$$

In our experiments, $d(\Delta P)$ is around 0.069 kPa (minimum ΔP applied by the flow controller), and the lowest S that we measured was of the order $O(10)$ μm (but not considered in the calculation of τ_{yM} as $10\ \mu\text{m} < d$). Thus, we assumed $d(\Delta P) = 0.069$ kPa and $dS = 10$ μm. We calculated uncertainty in the measurement of τ_y for the smaller tube $d_t = 127$ μm for a range of τ_y varying between 10^{-3} to 10^{1} Pa, and ΔP varying between 10^{-2} to 10^{3} kPa using Eqn. S45 as shown in Fig.S13. If we consider a yield stress with uncertainty of 10% to be sufficiently precise, then this 10% tolerance window narrows as τ_y decreases as shown in Fig.S13. Consequently, a smaller range of ΔP is available for conducting experiments to attain precise results at low τ_y. At higher ΔP, the uncertainty arises primarily from measuring a small S, while at lower ΔP values, it stems from the uncertainty in ΔP itself. In our experiments, we varied ΔP between $O(1)$ to $O(10^3)$ kPa, and thus, we could measure τ_y between 10^{-2} to 1 Pa with fair precision (~10%).

11. More on the limitations of the MEFD

11.1. The depth constraint in the MEFD

In the current setup, the flow in the depth direction is confined to $d = 117$ μm. If the test fluid contains large particles (as large as tens and hundreds of μm), they will obstruct the flow, and the measurement of τ_y cannot be performed. If we increase d up to a few mm or cm, it may be difficult to view the fluid flow at the center plane in the case of concentrated suspensions. But it turns out that the unyielded region not only occurs at the midplane, but it also occurs in the bottom or top plane, as shown in ESI Fig.S1C. We will explore this aspect in the future, particularly with the concentrated suspensions. This includes finding a separate characterization equation for the top or bottom plane, which needs to be determined before performing the measurements. Further, several studies demonstrate confinement effects on the flow of a yield stress fluid, typically towards reducing the yield stress.[21–24] There is a length scale associated with the microstructure within the material, termed as the cooperativity length scale. If the length of the confined dimension (in our case, $d = 117$ μm) is at least an order higher than the cooperativity length scale, then the confinement effects are not observed. For CS, the cooperativity length scale is of the order of 10 μm,[21–24] thus the confinement effects are unlikely to play a role in the MEFD. But this needs to be factored in while fabricating the MEFD.

11.2. Investigation of particle accumulation in the MEFD

In the past decade, considerable research has been dedicated to investigating shear banding in yield stress fluids, a phenomenon characterized by the segregation of sheared material into a high-strain band compared to the surrounding material.[25] This phenomenon is pervasive in complex materials and has been observed in systems such as carbopol gel and colloidal glassy materials.[26,27] It has been demonstrated that shear banding results in non-linear velocity profiles across the gap between the cone and plate in a rheometer, particularly below a critical shear rate $\dot{\gamma} = 0.2\ \text{s}^{-1}$.[26] This behaviour is attributed to changes in the concentration of particles across the sheared gap, which occur due to shear-induced migration towards regions of lower shear rate.[26] Given the spatially varying nature of shear rate in our MEFD rheometer, it is imperative to investigate the potential occurrence of shear banding, which could result in the accumulation of carbopol near the centre and consequently lead to a pseudo or apparent yield stress. To address this, we used images extracted from our experimental video and developed a MATLAB code to track the tracer particles. This code enabled us to measure the density of tracer particles across an 8×8 grid within the image, providing insights into the potential occurrence of any high concentration of particles near the centre of the MEFD. In Fig.S8A and B, we present both the experimental image and the corresponding analysed image of 0.3% CS at $\Delta P = 138$ kPa, with detected particles highlighted in red. In Fig.S8C and D, we depict the particle density distributions for 0.3% at ΔP of 138 kPa and 241 kPa, and similarly, for 0.05% at ΔP of 14 kPa and 34 kPa. Upon examination of these figures, it is evident that there is no discernible preferential crowding of particles closer to the centre of the MEFD. Instead, the particle density appears to be

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randomly distributed across all grid points. Therefore, it appears that there is no migration of particles, indicating that we are not measuring an apparent yield stress induced by increased concentration at the centre of the device.

Table S1: Herschel-Bulkley parameters listed for CS of C_{CS} ranging between 0.015 to 0.3% for rheological data shown in Fig.S3A.

Concentration of carbopol, C_{CS} (%)	Yield stress, τ_{yR} (Pa)	Consistency index, m_{pR}, $(\mathrm{Pa}-\mathrm{s}^{n_{fR}})$	Flow index, n_{fR}
0.3	1.2941	2.4935	0.49
0.2	0.9610	1.7401	0.49
0.1	0.5321	1.2204	0.48
0.05	0.0963	0.1260	0.61
0.025	0.0210	0.0210	0.83
0.015	0.0094	0.0034	0.99

Movie S1 (separate file). Flow of human blood (donor W_1) through the MEFD. We observe a relatively stagnant, non-moving region closer to the center, as demarcated by yellow box.

Movie S2 (separate file). Flow of 20% mucin suspension through the MEFD. We observe a relatively stagnant, non-moving region closer to the center, as demarcated by yellow box.

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