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Viscosity measurement of electrorheological fluids: corn starch and silicon microparticles mixed with silicone oil

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Abstract. A sensitive viscometer performing the shear-stress and shear-rate measurement of electrorheological fluid is constructed. This apparatus can measure the absolute value of the low viscosity of pure water as low as 0.001 P with an accuracy of 10% as calibrated from the standard value. The stress–strain relation of electrorheological fluids showing step-wise stick–slip response can clearly be observed from this instrument.

1. Introduction

An electrorheological (ER) fluid [1, 2] is a colloidal suspension of polarizable particles embedded in an insulating oil having a lower dielectric constant. Before applying the electric field, the fluid flows freely like a liquid. However, the particles begin to move under an electric field of several KV mm^{-1}. The dipole force for neighbouring particles along the direction of the field is attractive, while for those away from the axis with intersection angles greater than 55° it is repulsive. With the increase of the electric field, the particles link together to form thin chains which combine to form thick columns over several milliseconds, and the effective viscosity of the fluid is greatly increased [3]. At maximum electric field, the chains stick to the wall of the container, and the fluid becomes static like a solid. When the field is removed the chains are released and the quickly reversible liquid-to-solid transition with the correspondingly vast change of viscosity is the property that attracts many scientists' attention. Possible practical devices, based on the ER fluids, include variable-differential power-transmission clutches, variable-flow pumps, shock absorbers, and some robot control machines [4–6].

Since research into ER fluids is still developing, many measuring instruments are not available commercially. In this paper, a new shear-stress viscometer, developed to measure the absolute viscosity and shear force of ER fluids with high accuracy when applying the electric and magnetic fields, is described. Relevant properties of chain formation of these ER fluids were observed using an optical microscope to verify the change of the viscosities.

2. Theoretical background

Tao *et al* [3] briefly described how osmotic pressure becomes negative and the ER fluids become solid as the applied electric field exceeds some critical value E_c. This can be recognized from the thermodynamic view of the volume derivative of the free energy which consists of dipole interaction energies and thermal energies [7]. The interaction between induced dipoles in particles is attractive and favours the formation of chains along the direction of the electric field, while the thermal diffusion of the particle tends to randomize the particle positions. The initial field E_c is derived from the osmotic pressure which changes sign from positive to negative according to

$$E_c = (1 - n\bar{v})\sqrt{8\pi K T/\alpha^2 \bar{v}^2 n \varepsilon_f} \tag{1}$$

where n is the density number of dielectric particles, $\bar{v}$ is the thermal velocity of particles at temperature T, $\alpha = g/[g + \varepsilon_p/(\varepsilon_p - \varepsilon_f)]$, g is the particle geometry factor, and ε_f and ε_p are the dielectric constant of fluid and particles respectively. Equation (1) infers that the critical field depends on a complex quadratic function of the particle density n and decreases as n increases.

The high-dielectric particles are polarized in the presence of an electric field. The dipole–dipole interaction energy U has the well known form [8, 9]

$$U = \sum_{i,j} -\nu \left[2 + \rho \frac{\partial}{\partial \rho}\right] \frac{1}{r_{ij}^3} \tag{2}$$

where $r_{ij} = |r_i - r_j| = [\rho^2 + (z_i - z_j)^2]^{1/2}$ is the distance between dipoles at $\boldsymbol{r}_i$ and $\boldsymbol{r}_j$, $\rho = [(x_i - x_j)^2 + (y_i - y_j)^2]^{1/2}$,

$\nu = p^2/\varepsilon_f$ and $p = \alpha\varepsilon_f\beta^3 E_{loc}$ is the induced dipole moment under a local electric field E_{loc} and $\beta = (\varepsilon_p - \varepsilon_f)/(\varepsilon_p + 2\varepsilon_f)$. The internal energy U includes those particles arranged in the same chains and those in neighbouring chains in the same column which are given respectively as [8]

$$U_{chain} = -\frac{\nu}{4a^3}\sum_{s=1}^{\infty}\frac{1}{s^3} \tag{3}$$

and

$$U_i(\rho, z) = \frac{\nu}{a^3}\sum_{s=1}^{\infty} 2\pi^2 s^2 k_0(s\pi\rho/a)\cos\left(\frac{s\pi z}{a}\right) \tag{4}$$

where k_0 is the zero-order coefficient of modified Bessel functions, and $2a$ is the chain lattice constant.

A detailed calculation reveals that a body-centred tetragonal (bct) structure of the particles can yield a stable structure with minimum internal energy within one column of arrays. However, the stable macroscopic structures between columns has still not been successfully explored [10]. The chain formation for a stable structure after applying the electric field is the reason for the increasing viscosity of the ER fluid as studied in this work.

Another most intriguing feature of the ER fluid is that the shear stress for the chain structure, irrespective of multiple chains or bct lattice chains, suggests it is at its most stable when in a slanted configuration [11] where the shear strain is small and shows a stick–slip response when the electrode plates are moving but not parallel with each other. As the shear strain exceeds a yield point, the structure breaks into parts which cannot quickly return to the original configuration. The bct lattice has the strongest shear modulus, while the single-chain structure with the Pierls–Landau instability is weakest.

The shear strain δ/L, where δ is the slanted distance on the surface for each chain and L is the chain length, then the response force per chain is denoted by [8]

$$\tau = -\frac{\partial U(\delta)}{\partial\delta}. \tag{5}$$

Close examination [11] shows that the response force is linear with δ/L for $\delta < \delta_c$, and reduced to a small constant value for δ greater than that for the chain broken δ_c. This has been proven in this experiment.

Shearing stress builds up in an ER fluid when the electrodes start to move parallel with each other. This shear causes the chains to tilt and stretch without breaking and the ER fluid still has high viscosity. However, when the chains are tilted, the attractive force between particles is diminished, and the particles slip out of the zone. If the tilt is too great, the chains break and the viscosity suddenly decreases. The amount of stress at failure is called the yield stress. The broken chains may then recombine with neighbouring broken chains and the viscosity will increase. These theoretical properties of ER fluid will be tested by the viscometer described in this paper.

3. Experimental procedure

The ER materials illustrated in this work are silicon microcrystals and corn starch immersed in silicone oil. To produce Si microparticles, Si wafers of 1–10 Ω cm are ball milled for 1 h, and then baked at 250 °C for 1 h to remove moisture. The corn starch was ball milled for 2 h, and then baked at 105 °C for 3 h. Using a microscope, the particle sizes prepared by ball milling were seen to be approximately 10 μm. The particles were then mixed with silicone oil at a predetermined percentage by weight and placed between two transparent glass plates. The inner surfaces were coated with indium–tin-oxide (ITO) using ion sputtering. A teflon or viton O-ring, 1 mm thick was used to retain the ER fluid between the glass plates and the whole assembly was held together by two acrylic plates.

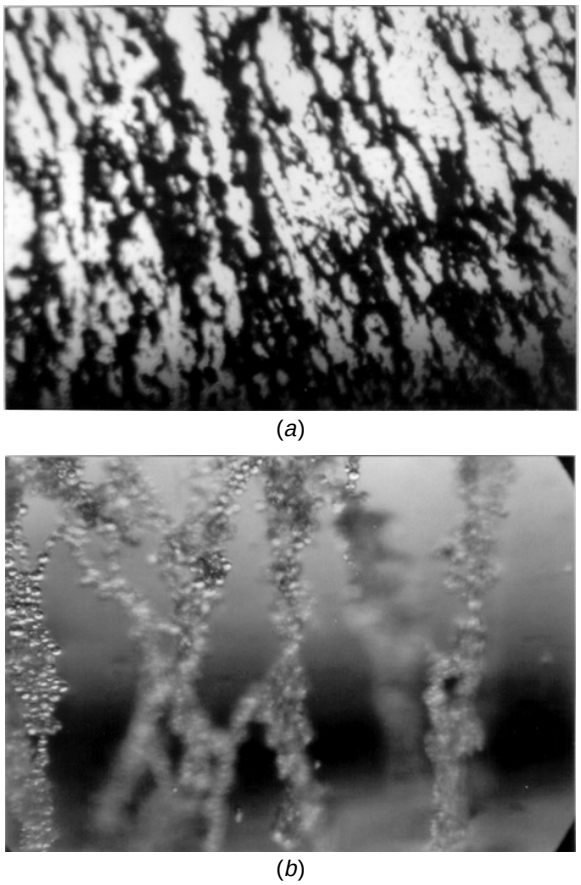
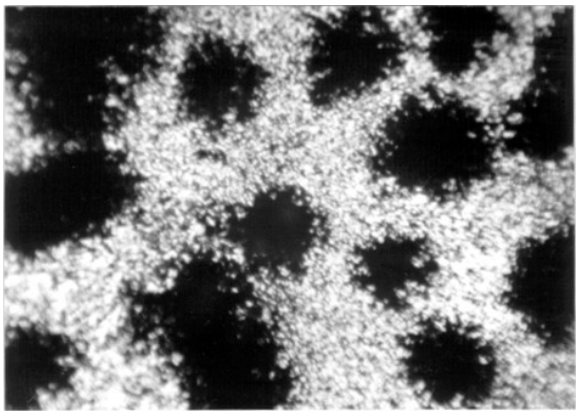


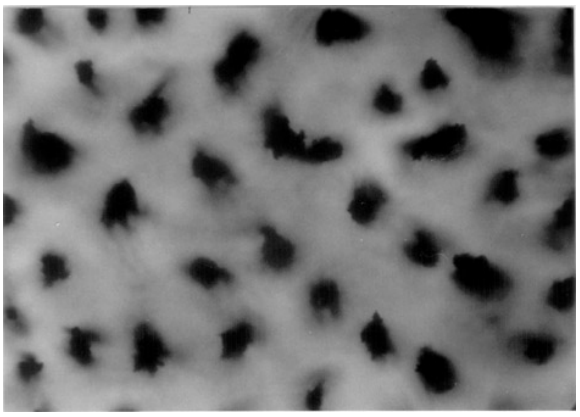
Figure 1. Side-view photographs showing the chain formation of ER fluid under an electric field for (*a*) silicon powder mixed with silicone oil at 0.5 Wt% under E = 300 V mm^{-1}, and (*b*) corn starch mixed with Si oil at 0.5 Wt% under E = 900 V mm^{-1}, respectively.

Figure 1(*a*) shows the chain formation for the ER fluids consisting of 0.5 Wt% silicon powder and figure 1(*b*) that for corn starch, with silicone oil, at an electric field of 900 V mm^{-1}. Viewing the two fluids from above (see figures 2(*a*) and (*b*)), the ER fluid consisting of 1 Wt% Si powder, shows almost regular columns. Although the stable lattice structure is well explored in [8] the regular column features are still not understood.

In order to measure the shearing-stress response of the ER fluids, a slow shear-rate viscometer was constructed as shwon in figure 3(*a*) while the actual instrument is photographed in figure 3(*b*). Other viscometers have been described elsewhere [12, 13]. In this construction,



(*a*)



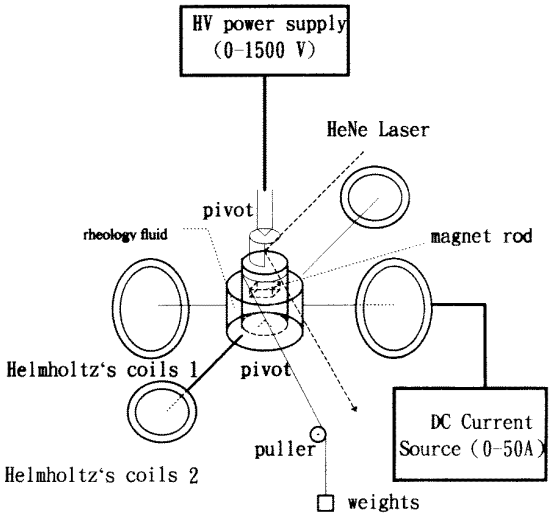
(*b*)

Figure 2. Top-view photographs of the column formation of ER fluid (*a*) silicon powder at 1 Wt% under E = 1000 V mm^{-1}, and (*b*) silicon powder at 0.2 Wt% under E = 1000 V mm^{-1}, respectively.

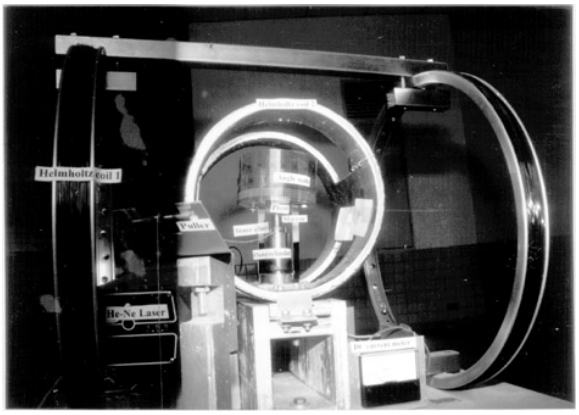
we can align the inner cylinder easily by applying two perpendicular Helmholtz coils, and measure the absolute value of viscosity by a free-fall weight coupled to a pulley. The final constant velocity of the falling weight is calculated from the time interval falling through a constant distance. Owing to the high viscosity of the ER fluid, constant falling velocity is arrived at within a few seconds of starting the experiment.

The method of carrying out the experiment is now described. The ER fluid is poured into the space between two concentric stainless cylinders, the bottoms of which are fitted with teflon sheets pivoted at the centre, allowing the inner cylinder to rotate smoothly with little friction. A permanent magnet is laid horizontally inside the inner cylinder. At the start of the experiment the auxiliary Helmholtz coil (2) will rotate the magnet perpendicular to the axis of the main Helmholtz coil (1). The magnetic field in the Helmholtz coil is constant over a large area, and, therefore, can readily be measured by a Hall gaussmeter. When in this position, the torque applied on the inner cylinder is maximum by the main Helmholtz coil.

The top of the inner cylinder has a pivot connected to the high voltage providing a low-friction bearing. Below the pivot, a small mirror is attached to reflect the He–Ne laser beam when the main Helmholtz coil is energized.



(*a*)



(*b*)

Figure 3. (*a*) Schematic diagram of a slow shear-stress shear-rate viscometer. (*b*) Photograph of the actual instrument.

This deflection angle and the magnetic field can respond to the strain and shear stress respectively. The angular resolution of the laser deflection can be as accurate as 1 mm $(2\text{m})^{-1} = \pi \times 10^{-3}$ rad. To measure the absolute viscosity of the ER fluid, a thin silk wire is wound around the upper surface of the inner cylinder; the end of the wire, with a weight attached, then passes over a pulley. The falling velocity of the weight measures the absolute value of the viscosity.

4. Shear-stress and strain measurement

We can express the shear stress under a strain by the formula [14, 15]

$$\sigma(E, \dot{\gamma}) = \sigma_y(E) + \eta(E)\dot{\gamma} \tag{6}$$

where $\sigma(E, \dot{\gamma})$ is the shear stress of the ER fluid under an electric field E at a shear rate $\dot{\gamma}$ which is defined as $\mathrm{d}u/\mathrm{d}y$, the derivative of fluid velocity $\boldsymbol{u}$ with respect to the displacement perpendicular to $\boldsymbol{u}$, $\eta(E)$ is the viscosity of the fluid, and $\sigma_y(E)$ is the yield shear stress. The

Bingham ER fluid as shown by equation (6) is reduced to a Newtonian fluid $\sigma_y(E) = 0$.

In this viscosity measurement, there is a background torque τ_0 arising from the shear stress applied by the ER fluid with zero field on the inner cylinder which can be determined from the deflection angle θ at a magnetic field B (by the main Helmholtz coil), i.e.

$$\tau_0 = \mu B_1 \cos\theta_1 = \mu\alpha_1 \tag{7}$$

where θ_1 is the deflection angle of the inner cylinder from its original direction (i.e. along the axis of the auxiliary Helmholtz coil). Since the absolute value of the permanent magnetic dipole μ is unknown, therefore τ_0 must be determined. We can put a weight F at the end of the silk wire. The weight will start to rise, if the magnetic field B_2 is high enough. The force equilibrium yields

$$(F + F_0)R + \tau_0 = \mu B_2 \cos\theta_2 = \mu\alpha_2 \tag{8}$$

where F_0 is the friction force from the pulley and the wire with the surface of inner cylinder, R is the radius of the inner cylinder and θ_2 is the deflection angle at this equilibrium condition. Using the same weight, a decrease in the magnetic field to B_3 will cause the weight to fall. In the same way, we have

$$(F - F_0)R - \tau_0 = \mu B_3 \cos\theta_3 = \mu\alpha_3. \tag{9}$$

From these three equations, we can solve

$$\mu = 2FR/(\alpha_2 + \alpha_3) \qquad \tau_0 = 2FR\alpha_1/(\alpha_2 + \alpha_3)$$

and

$$F_0 = \frac{(-2\alpha_1 + \alpha_2 - \alpha_3)F}{\alpha_2 + \alpha_3}. \tag{10}$$

Figure 4 indicates the experimental result for the ER fluid made of corn starch at 5 Wt%. Initially, the deflection angle is small. When the magnetic field increases above 10.05 Gauss (B_2), the weight is pulled up with a pronounced deflection angle θ_2. During this process, the $B_2 \cos\theta_2 = \alpha_2$ remained almost constant with an average value of $\alpha_2 = 11.36$ Gauss. By decreasing the magnetic field, the weight begins to fall appreciably at $B_3 = 5.43$ Gauss. The calculated value of $\alpha_3 = B_3 \cos\theta_3$ is nearly 4.33 Gauss for a magnetic field smaller than B_3. This experiment can be repeated with almost the same result.

Now we return to equation (6), the shear stress is

$$\sigma_y(E) = F/A = \frac{\tau - \tau_o}{AR} = \frac{\mu}{2\pi LR^2}(B\cos\theta - B_1\cos\theta_1) \tag{11}$$

where A is the area of the fluid inner surface. This experiment (as shown in figure 5) exhibits a linear electric dependence of $B\cos\theta$ for the ER fluid at a low field (i.e. $B\cos\theta = 0.005\,102E + 3.385$), which, using equation (8), $\sigma_y(E)$ can be solved for corn starch ER fluid as

$$\sigma_y(E) = (0.0030E + 0.6985)\ \text{N m}^{-2}. \tag{12}$$

The nonlinear dependence of σ_y on E may occur at high electric field. Applying the electric field to the ER fluid, a step-wise deflection angle, with respect to the magnetic field, can be observed from figure 6. The stress strain indicated is almost constant before the chains inside the fluid break. At this point the ER fluid resists the shear torque preventing the inner cylinder from rotating.

With a small increase in stress (caused by the applied magnetic field), a stick–slip shear will cause the chain to break, and the fluid will begin to flow. This shows that this material does yield a stick shear stress, and that it sustains a significant shear until the yield stress is exceeded. The drain

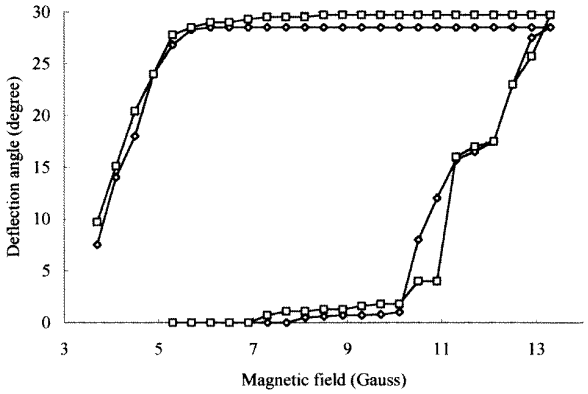


Figure 4. The shear-strain (the deflection angle) and the shear stress (the magnetic field) response of the Er fluid consisting of corn starch at 5 Wt% and silicone oil. Symbols ◊ and □ represent the first and second trials of the experiments which are in an anticlockwise sequence.

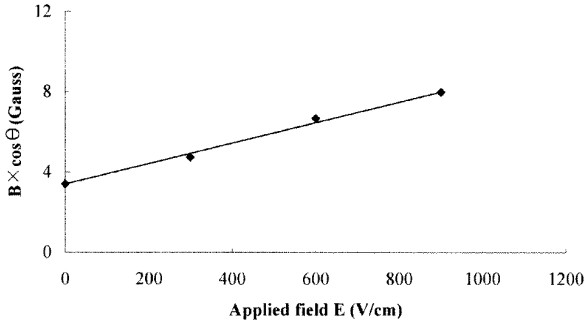


Figure 5. The linear electric dependence on $B\cos\theta$ for the ER fluid at low electric field.

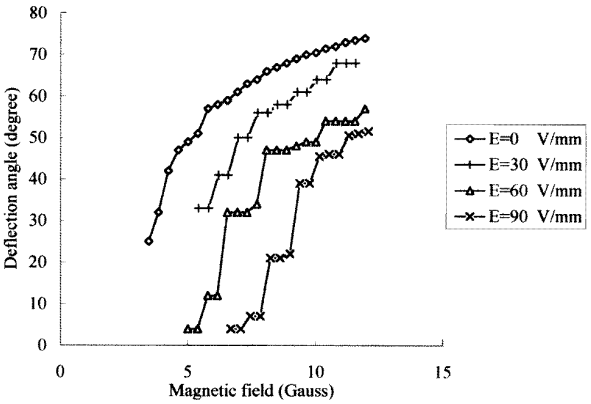


Figure 6. Step-wise deflection of the corn starch ER fluid showing the stick–slip shear changes due to chain formations, deformation broken and recrystallization.

connection of the particles recovers after some displacement and the stick stress repeats again.

5. Absolute values of viscosity

Assuming that the inner cylinder rotation inertia is I the dynamic friction of its pivots is β_h, and the dynamic friction of the pulley is α_k, we can determine these three parameters, without using any fluid, by hanging three different weights, m_i, at the end of the wire and measuring their angular acceleration velocities, $\dot{\omega}_i$, which follows $\dot{\omega} = 2\theta/t^2 = 2h/Rt^2$ and

$$(m_i g - \alpha_k m_i g - \beta_k)R = I\dot{\omega}_i. \tag{13}$$

If pure water is then placed into the container, the equations of motion change to

$$\left(mg - \alpha_k mg - \beta_k - \eta\frac{\mathrm{d}u}{\mathrm{d}y}A\right)R = I\dot{\omega} \tag{14}$$

where η is the viscosity of water ($\eta = 0.001$ P at 20 °C, 1 atmosphere), $\mathrm{d}u/\mathrm{d}y = R\omega/\alpha$ is the flow velocity which is a function of the rotation speed of the inner cylinder, and the buoyancy of water B_w, which reduces the pivot friction of the inner cylinder is negligibly small compared with other terms. Also $B_w = \pi R^2 L\rho$, where L is the container length, R is the radius of inner cylinder and ρ is the density of water. The above equation implies that the angular velocity will approach a constant when the observing time is much greater than $(2\pi R^3\eta L/Id)^{-1}$. For the ER fluid, the fall time to reach constant final velocity will be much shorter.

On exchanging the ER fluid for the water, the time to reach a constant falling velocity is shorter, and the shear stress is

$$\sigma = \frac{mg - \alpha_k mg - \beta_k}{2\pi RL} \times 10^{-1}\ \mathrm{N}\ m^{-2}$$

and the shear rate is

$$\dot{\gamma} = \frac{\mathrm{d}\eta}{\mathrm{d}y} = \frac{\text{constant velocity}}{d}\ \mathrm{s}^{-1} \tag{15}$$

This shear stress versus shear rate at different electric fields is plotted in figure 7.

6. Conclusion

From this experiment, we find the particles inside the ER fluid flow continuously after applying the electric field and stop after a few seconds. With a higher electric field, the motion is faster and much more regular. On removal of the electric field, the ER particles do not return to their random locations. Therefore, the ER fluid is not reversible for a static fluid. Only in the dynamic motion (i.e. with shear rate $\dot{\gamma} \neq 0$), the shear stress reacts 10^{-3}–10^{-2} s after applying the field and then the random/regular arrangement of the ER particles is reversible. This instrument can measure the absolute value and small change of viscosity of many ER fluids during application of the electric field. Fascinating features of ER fluids showing step-wise stick–slip response can clearly be observed from this instrument. The accuracy of the velocity measurement can be greatly improved by using stroboscopic photography to measure the falling speed of the weight which is pulled by the ER fluid.

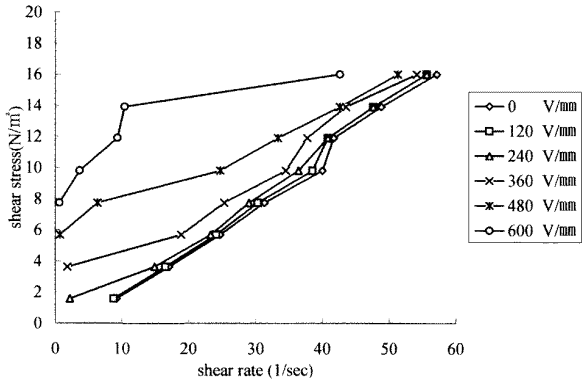


Figure 7. The shear stress versus shear rate of corn starch ER fluid at different electric fields.

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