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Reduction of clean fracturing fluid filtration loss by viscosity enhancement using nanoparticles: Is it feasible?

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ABSTRACT

Fracturing fluid filtration loss is a significant research topic related to fracture propagation, formation damage and the cost of hydraulic fracturing. To reduce the loss, nanoparticles have been used to enhance the fracturing fluid properties such as viscosity and well-building ability. In this work, silica nanoparticles (NPs) with different concentrations (0, 0.01, 0.03 wt%) were added into the viscoelastic surfactant (VES) fluid composed of cetyltrimethylammonium bromide (CTAB) and sodium salicylate (NaSal). A micro pore-throat structure was applied to investigate the flow characteristics of those three fracturing fluids with Deborah numbers (*De*) spanning from 1.52 to 73.2, and a Newtonian fluid was set as the control group. It was found that the flow patterns of VES solutions performed as the convergent flow similar to those of the Newtonian fluid at upstream, and turned into divergent flow with the gathering of streamlines at downstream. Surprisingly, the flow characteristics between VES solution with no nanoparticles and nanoparticle-enhanced the VES solution showed an insignificant difference with high flow rates (5 and 20 ml/h). The nanoparticle was unable to enhance or influence the properties of VES fluid when flowing through convergence–divergence and divergence–convergence structure. Neither the breakage nor the recombination of wormlike micellar were barely affected by the nanoparticle.

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1. Introduction

Viscoelastic surfactant (VES) fluid has been applied diffusely in cosmetics stabilization, pharmaceutical production and food processing (Rehage and Hoffmann, 1988; Zhao et al., 2016). In especial, VES fluid recently draws great attention in oil and gas recovery as fracturing fluid due to its controllability of gelling and breaking, with no residue and no formation damage (Arnippally and Kuru, 2017; Liu et al., 2019; Smirnov

et al., 2006; Osborn et al., 2011; Vengosh et al., 2014; Al-Muntasheri et al., 2017). The viscoelastic of polymer solutions, such as hydrolyzed polyacrylamide (HPAM) solution, come from the entanglement of the long chain molecules, while the increasing viscosity of VES fluids is generated by the assembled wormlike micelles entangling with each other (Zhao et al., 2016). As a result, the formation damage caused by VES fluids is smaller than that of the polymer fracturing fluids, and the stability of VES fluids is higher. However, due to the breakage and recombination of wormlike micellar under strong elongation and shear

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flow, the high leak-off property of VES fluids is a significant concern (Samuel et al., 2000).

To overcome this drawback, nanoparticles were applied. It was found that nanoparticles could enhance the rheological properties of VES fluids and form filter cakes to reduce filtration loss (Petros and DeSimone, 2010; Qu et al., 2013; Alaskar et al., 2012; Shen et al., 2008; Cai et al., 2012). Huang and Crew investigated the rheological properties of amidoamine surfactant with 30-nm ZnO nanoparticles (Huang and Crews, 2008). Their study indicated that surface charge attraction and adsorption of the VES fluid on the nanoparticles played a key role in the viscosity enhancement. Spinning from 1 to 100 nm, nanoparticles could provide huge surface areas for adsorption. Recently, more and more attractions have been paid to enhancing the performance of VES fluids with the addition of silica nanoparticles (Zhang et al., 2016; You et al., 2012). The ability of nanoparticles to increase the relaxation time of wormlike micelle solution composed of cetyltrimethylammonium bromide (CTAB) and sodium nitrate (NaNO_3) has been proven by Nettesheim et al. (2008). Experimental results of Dai et al. (2017) also showed that the viscosity enhancement was apparent at low shear rates (lower than 1 s^{-1}) with the addition of nanoparticles to a VES fluid (CTAB/sodium salicylate (NaSal)). The mechanism lies in the newly formed micelle-nanoparticle junctions to improve the viscoelasticity. Besides increasing rheological properties, Huang et al. (2010) concluded that nanoparticles were able to reduce the filtration loss effectively by strengthening the well-building properties as well. The filtration loss data of Bybee (Bybee, 2009) found that the nanoparticle-pseudo crosslinked VES micelle fluid developed a wall-building pseudo filter cake on the face of porous media to control fluid loss.

However, the mentioned experiments were conducted by core experiments and commercial rheometers. The flow characteristics in micro pore-throat structures are unclear. As known, the flow characteristics have a significant effect on the filtration of fracturing fluids in porous media (Smirnov et al., 2006), which may be changed by nanoparticles and required further study. Microfluidic devices allow observing the flow behaviors in micro pore-throat structures directly (Wu et al., 2019; Dubash et al., 2012; Santiago et al., 1998; Wu et al., 2015; Frounfelker et al., 2009). Flow patterns of wormlike micelle solution in a microchannel were found to be able to form a stable flow-induced structured phase (FISP) (Cheung et al., 2012). Three VES systems (CTAB/hydroxy salt 3-hydroxy naphthalene-2-carboxylate (SHNC), CTAB/NaSal and cetylpyridinium chloride (CPyCl)/NaSal) were studied by Zhao et al. (2016) and Sun and Huang (2016) in straight microchannels with different blockage ratios of a cylinder. In their work, both extensional and shear strain rates were proven to contribute a flow-induced birefringence.

Former works of VES fluids indicated that both rheological properties and well-build properties were enhanced by nanoparticles to reduce the filtration. However, it is insignificant to figure out the effect of nanoparticle on enhancement of flow characteristics. In this paper, a core experiment was conducted firstly to study the filtration loss of VES fluid with no nanoparticle VES fluids and two nanoparticle-enhanced VES fluids. Afterwards, a micro pore-throat structure was fabricated and used to capture the flow characteristics in porous media. The

flow patterns of all solutions were discussed to determine whether the reduction of filtration is related to the enhancement of flow patterns by nanoparticles.

2. Materials and experimental methods

2.1. Experimental parameters and dimensionless groups

All the experimental parameters are listed in Table 1. There were two dimensionless numbers used in our work to quantify the flow field observed in micro pore-throat structure. Reynolds number (Re) is defined as (Li et al., 2019):

$$Re = \frac{\rho \bar{V}_p D_h}{\eta_p} = \frac{2\rho Q}{(w_p + h)\eta_p} \quad (1)$$

Here, $\bar{V}_p$ is the average flow velocity in a pore channel, $D_h = 2w_p h / (w_p + h)$ is the hydraulic diameter.

Deborah number (De) is the ratio of a solution's relaxation time to residence time in the flow as (Kenney et al., 2013):

$$De = \frac{\lambda_1}{w_p l h / Q} = \frac{\lambda_1 Q}{w_p l h} \quad (2)$$

2.2. Filtration loss experiment

Dynamic filtration loss experiments were conducted by the core experiment method. All cores were saturated with brine water for 24 h. After saturation, the core was placed in a core holder, and the confining pressure of the core holder was set as 6 MPa. The flowlines of the core experiment device were filled with water. The injection pressure was 3.6 MPa when the fracturing fluid passing through the core. The complete test was performed within 36 min. The filtration time and filtration volume were recorded when the first trickle filtrate flowed out. The core properties are listed in Table 2.

2.3. Microchannel design and fabrication

The microfluidic device was designed on a plate ($120 \times 100 \times 8 \text{ mm}$) of polymethylmethacrylate (PMMA) by precisely sealed with another PMMA plate ($120 \times 100 \times 2 \text{ mm}$). It consisted of an injection point, an outlet point, a liner microchannel and a continuous pore-throat structure (Fig. 1a). The continuous pore-throat structure possessed a contraction–expansion structure at upstream and an expansion–contraction structure at downstream (Fig. 1b), and had a uniform depth of $100 \mu\text{m}$. A syringe (25 ml, Hamilton) driven by a syringe pump

Table 1 – Experimental parameters.

Q (ml/h)	Flow rate	w_p (μm)	Width of pore-throat
w_c (μm)	Width of microchannel	l (μm)	Length of pore-throat
h (μm)	Depth of microchannel	ρ (g/cm^3)	Solution density
D_h (μm)	Hydraulic diameter	Re	Reynolds number
η_s (mPa s)	Solvent viscosity	η_p (mPa s)	Shear-viscosity
λ_1 (s)	Relaxation time		

Table 2 – Core properties.

Core no.	Fluid	Length (mm)	Diameter (mm)	Porosity (%)	Permeability (mD)
1	VES	49.30	25.34	21.36	7.24
2	VES +0.01 wt% NPs	48.88	25.27	21.27	7.59
3	VES +0.03 wt% NPs	49.93	25.28	21.52	7.73

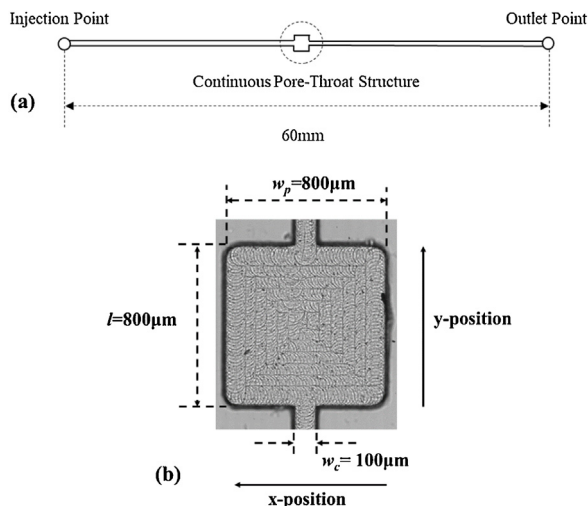


Fig. 1 – (a) Layout design of the microfluidic device. (b) Geometric parameters of the continuous pore-throat structure.

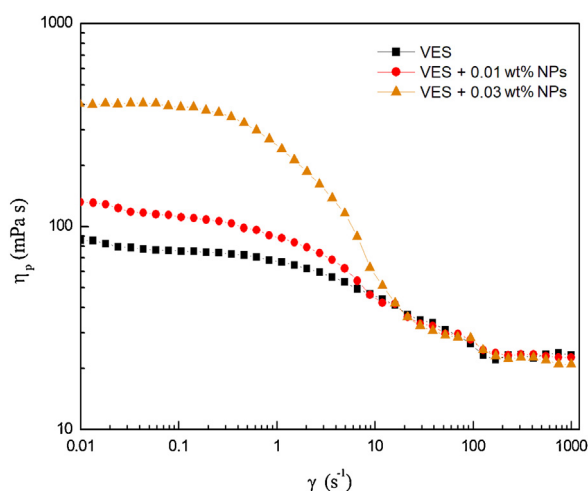


Fig. 2 – Shear-viscosity η_p of the VES solutions as shear rates γ rising from 0.01 s^{-1} to 1000 s^{-1} .

(PHD/ULTRA, Harvard) was connected to the inlet point to obtain accurate and stable flow rates.

2.4. Fluid rheological characterizations

Three VES solutions with different concentrations (VES, VES +0.01 wt% and VES +0.03 wt%) of silica nanoparticles (LUDOX®, HS-30 colloidal silica) were used while a Newtonian solution (63 wt% glycerol–water solution) was selected as the control group. The VES solutions composed of 20 mMol/l CTAB and 10 mMol/l NaSal. The rheological characteristics were tested by HAAKE MARS 60 rheometer. The rheology measurement result was shown in Fig. 2. Due to the rheological characteristics of shear thinning, the viscosities (η) of both solutions were similar while shearing rates $\gamma > 10 \text{ s}^{-1}$. The relaxation times λ_1 were 0.35, 0.57 and 0.84 s for VES fluid, VES +0.01 wt% NPs and VES +0.03 wt% NPs.

A micro-particle image velocimetry (μ -PIV) was used to measure quantitative flow fields in the continuous pore-throat structure. The flow visualization followed the same method with our former work (Li et al., 2019).

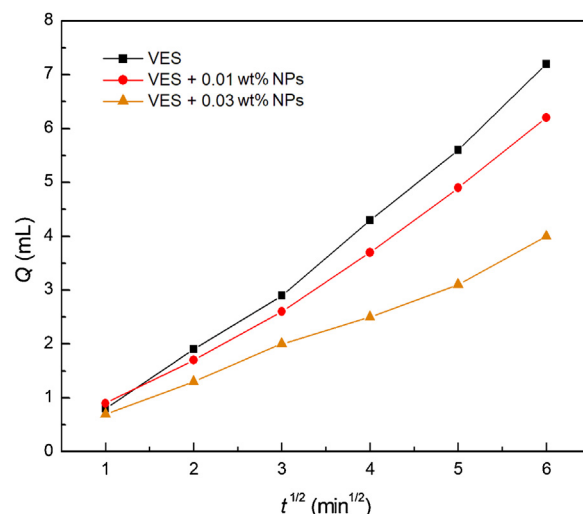


Fig. 3 – Filtration loss curves of VES, VES +0.01 wt% NPs and VES +0.03 wt% NPs.

3. Results and discussions

3.1. The filtration loss in the core experiment

The experimental filtration coefficient C (petroleum and natural gas industry standards SY/T 5107-2016) is calculated by Eq. (3), where m is the slope of filtration curve and A is the acreage of the core:

$$C = 0.005 \times m/A \quad (3)$$

As shown in Fig. 3, the slopes of filtration loss curves are 1.271, 1.063 and $0.642 \text{ mL/min}^{1/2}$, respectively. The filtration coefficients C are $1.26 \times 10^{-3} \text{ m/min}^{1/2}$ for the VES fluid, $1.06 \times 10^{-3} \text{ m/min}^{1/2}$ for the VES +0.01 wt% NPs fluid and $6.24 \times 10^{-4} \text{ m/min}^{1/2}$ for the VES +0.03 wt% NPs fluid. It is quite obvious that adding nanoparticles into fracturing fluids can reduce filtration loss. Fig. 4 shows the surfaces of two cores captured by SEM (scanning electron microscopy). Comparing Fig. 4(a) and (b), the nanoparticles in VES fluid +0.03 wt% NPs fluid adsorbed on the surfaces of core. This result accord with Dai's report (Dai et al., 2017) that the formation of a nano-filter cake can decrease the amount of VES fracturing fluid loss during filtration.

3.2. Microfluidic experiment

The core experiment has proven that filtration loss control can be improved by adding nanoparticle into VES fluids. However, classical filtration assessment in core experiments are achieved by analyzing macroscopic information, with very little information on the pore-scale mechanisms. It is unclear whether this reducing effect results from the enhancement in rheological properties of VES fluids by nanoparticles. In this section, the velocity fields of three VES fluids with different nanoparticle concentrations in micro pore-throat structure were observed by μ -PIV to clarify the effect of nanoparticle-enhanced rheological properties on the flow patterns.

3.2.1. Newtonian flow in microchannels

Flow patterns of the Newtonian fluid are first examined in pore-throat structure as the control group (Fig. 5). Its performance followed the same predicted behavior with our

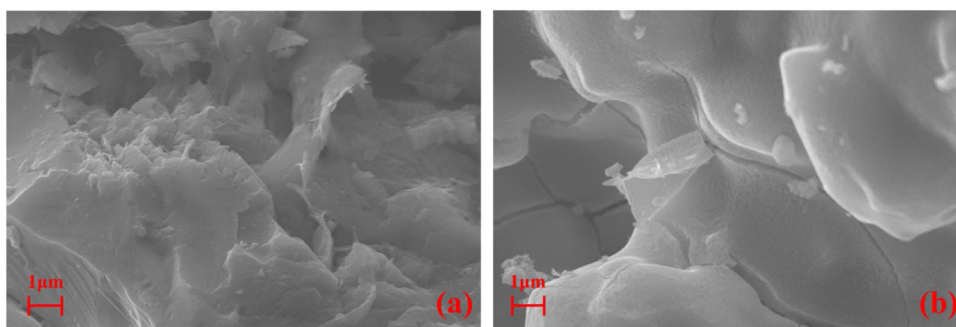


Fig. 4 – Adsorption of nanoparticles on the surfaces of (a) Core No.1 with VES fluid and (b) Core No.3 with VES +0.03 wt% NPs fluid.

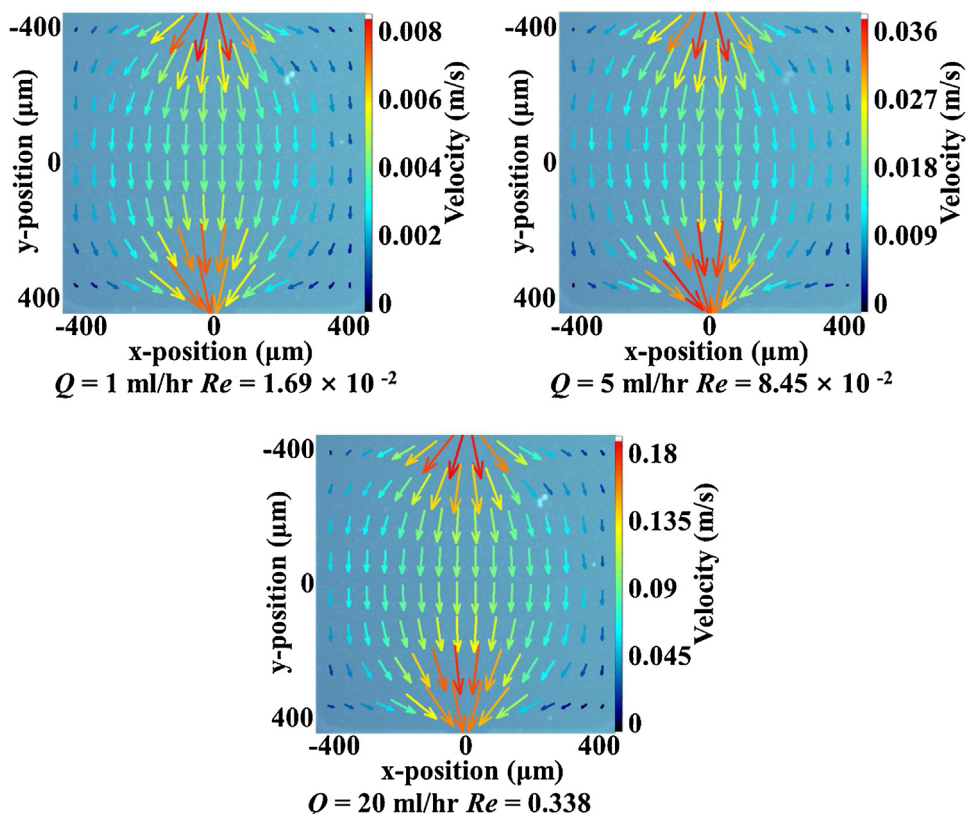


Fig. 5 – The flow patterns for the flow of Newtonian fluid in pore-throat structure, with flow rates of 1, 5 and 20 ml/h, and with $Re = 1.69 \times 10^{-2}$ to 0.338.

previous work (Li et al., 2019). Initially, flow patterns of the Newtonian fluid are symmetric at the upstream and downstream of the pore-throat structure. The maximum velocities are observed at the entrance and the exit of the pore-channel due to the gathering of streamlines. In the middle part of the pore-throat structure, the velocity component in the x-direction is negligible, from $y = -150$ to $150 \mu\text{m}$. Secondly, the velocity fields of the Newtonian fluid with different flow rates have similar flow patterns (Re number increasing from 1.69×10^{-2} to 0.338). Namely, flow patterns are unchanged with the rising flow rate.

3.2.2. Flow characteristics of the VES fluid with no nanoparticle in pore-throat structures

To investigate the flow characteristics of the VES fluid without nanoparticle, velocity fields of VES fluid in the pore-throat structure are illustrated in Fig. 6 at $Q = 1, 5$, and 20 ml/h . The flow characteristics show a significant distinction with the Newtonian fluid in Fig. 5.

Unlike the relatively divergent streamlines of the Newtonian fluid, the flow patterns of VES fluid present as a more contractive type. At low De number ($Q = 1 \text{ ml/h}$), the contraction behavior of streamlines is obvious at both upstream and downstream of pore-throat structure, gathering within the range from $x = -330 \mu\text{m}$ to $x = 300 \mu\text{m}$. While flowing through the joint of pore-channel and throat channel (nearly $y = -380 \mu\text{m}$), the velocity component along y-direction is not as large as the Newtonian fluid. Garcia-Sandoval et al. investigated the rheological properties of VES fluids, and found that the flow-induced breakage of wormlike micellar under shear and extend deformation experienced three microstates, from entangled wormlike chains to free wormlike chains to free short rod-like micelles (Paulo García-Sandoval et al., 2018). Therefore, the VES fluid still represents a certain structural viscosity at low De number, causing the mentioned contraction behavior of streamlines at upstream.

However, wormlike micellar breaks into free wormlike chains or free short rod-like micelles at $Q = 5$ and 20 ml/h ,

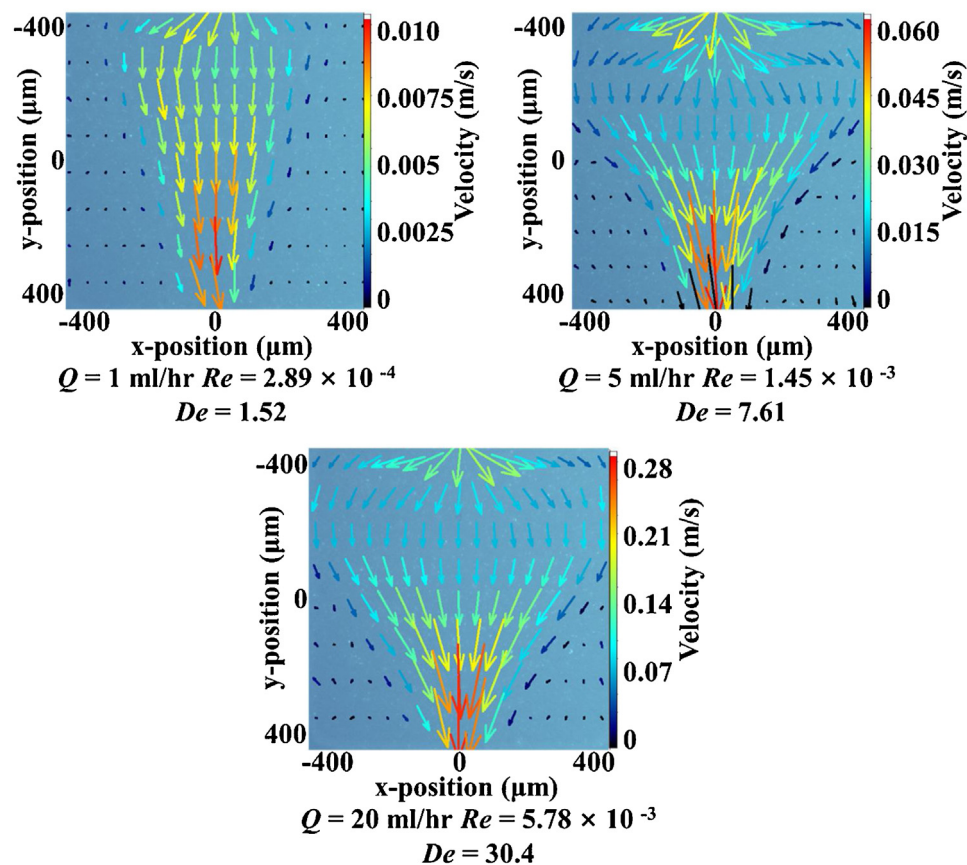


Fig. 6 – The flow patterns for the flow of VES fluid in pore-throat structure, with flow rates of 1, 5 and 20 ml/h, and with $Re = 2.89 \times 10^{-4}$ to 5.78×10^{-3} .

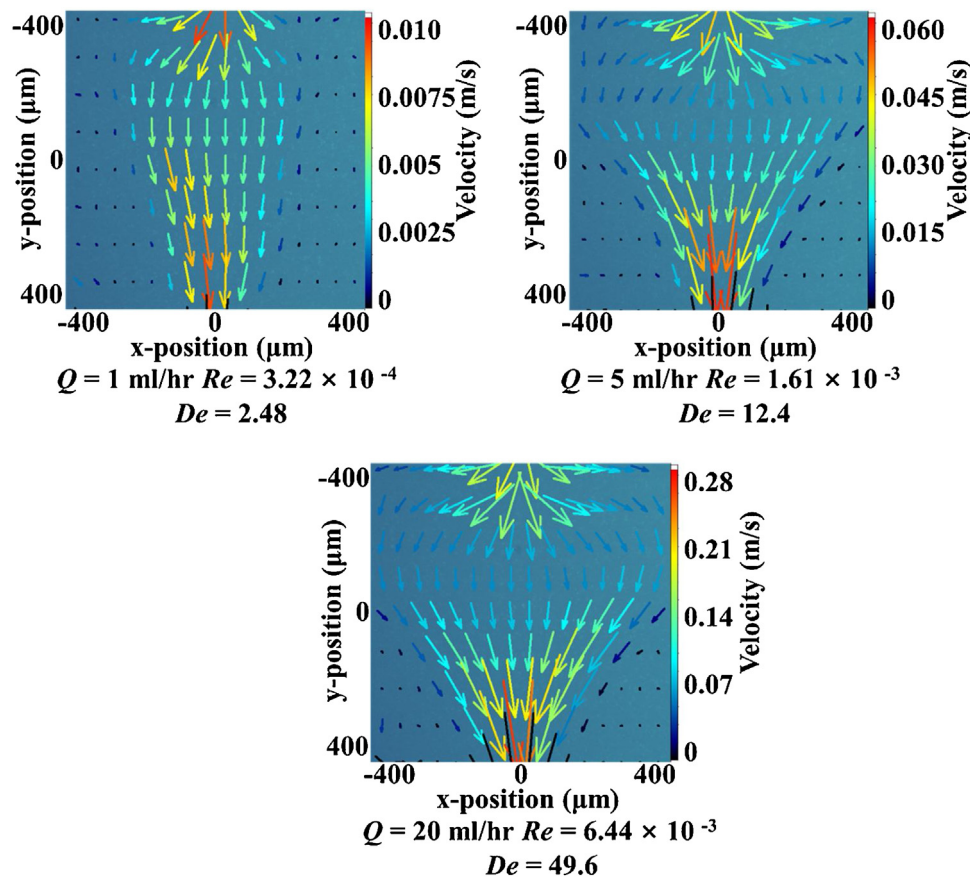


Fig. 7 – The flow patterns for the flow of VES +0.01 wt% NPs fluid in pore-throat structure, with flow rates of 1, 5 and 20 ml/h, with $Re = 3.22 \times 10^{-4}$ to 6.44×10^{-3} .

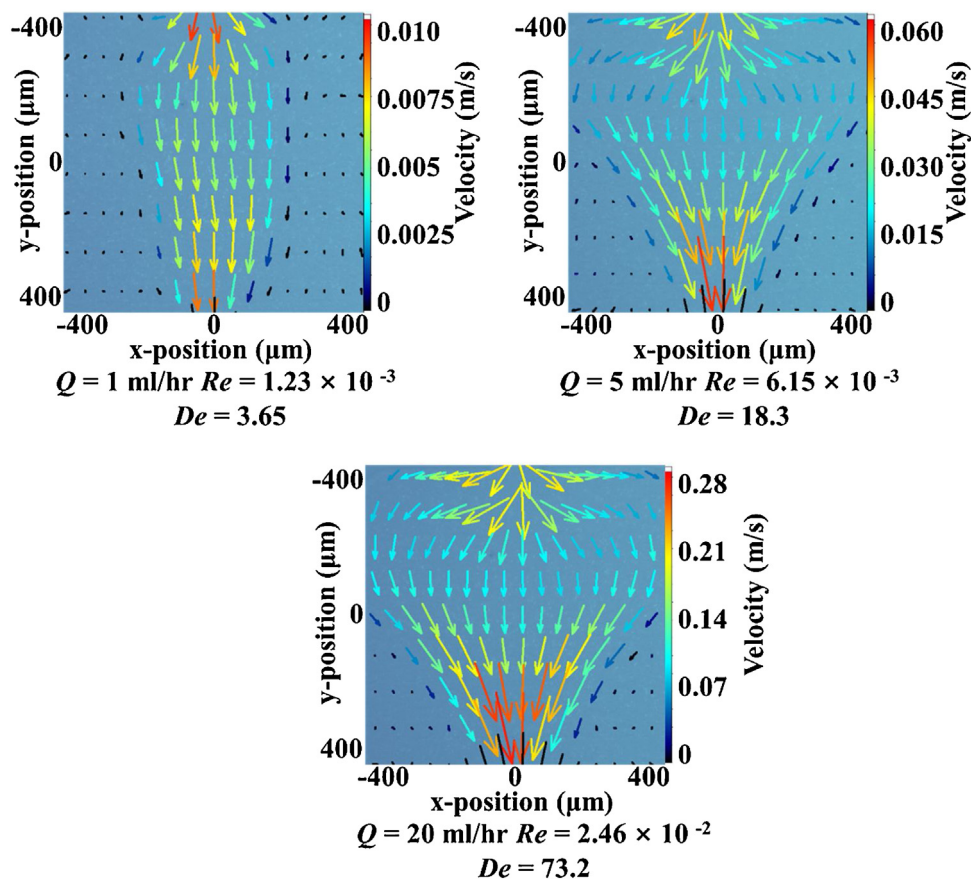


Fig. 8 – The flow patterns for the flow of VES +0.03 wt% NPs in pore-throat structure, with flow rates of 1, 5 and 20 ml/h, with $Re = 1.23 \times 10^{-3}$ to 2.46×10^{-2} .

leading to abrupt decreasing of viscosity. Consequently, the flow pattern at upstream transforms from the contraction behavior to the Newtonian-like characteristic. Velocity component along y-direction is much larger comparing with $Q = 1$ ml/h and occupies the dominant position of the flow. Streamlines reappear as divergent behavior and expand to the boundary ($x = -400 \mu\text{m}$ and $400 \mu\text{m}$). After that, the flow pattern follows the Newtonian-like characteristic before getting re-contractive around $y = -150 \mu\text{m}$. This contractive pattern persists until it reaches the exit of the pore-throat structure. In general, for $Q = 5$ and 20 ml/h, the flow patterns of VES fluid compose of Newtonian-like divergent behavior and viscoelastic contractive behavior. The symmetrical streamlines of the Newtonian fluid shown in Fig. 5 cannot be observed for VES fluid in Fig. 6.

3.2.3. Flow characteristics of nanoparticle-enhanced VES fluid in pore-throat structures

Two nanoparticle-enhanced VES fluids (VES +0.01 wt% and VES +0.03 wt%) are used to figure out the effect of flow characteristics modification and results are displayed in Figs. 7 and 8. To match the velocity in Fig. 6, injection rates applied in this section were set as 1, 5 and 20 ml/h as well.

Firstly, flow patterns still represent the mentioned contractive behavior at a low flow rate ($Q = 1$ ml/h). It accords with the rheological characteristics illustrated in Fig. 2 that nanoparticle is able to enhance the resistance of VES fluid against shear. Structural viscosities of two nanoparticle-enhanced VES fluids are theoretically larger than that of VES fluid without nanoparticle. Hence, this contractive behavior is reasonable.

Nevertheless, the enhancement of nanoparticle on the structural strength of VES fluid is not significant at higher flow

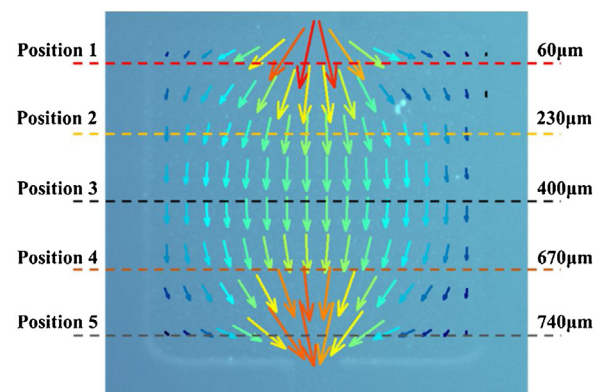


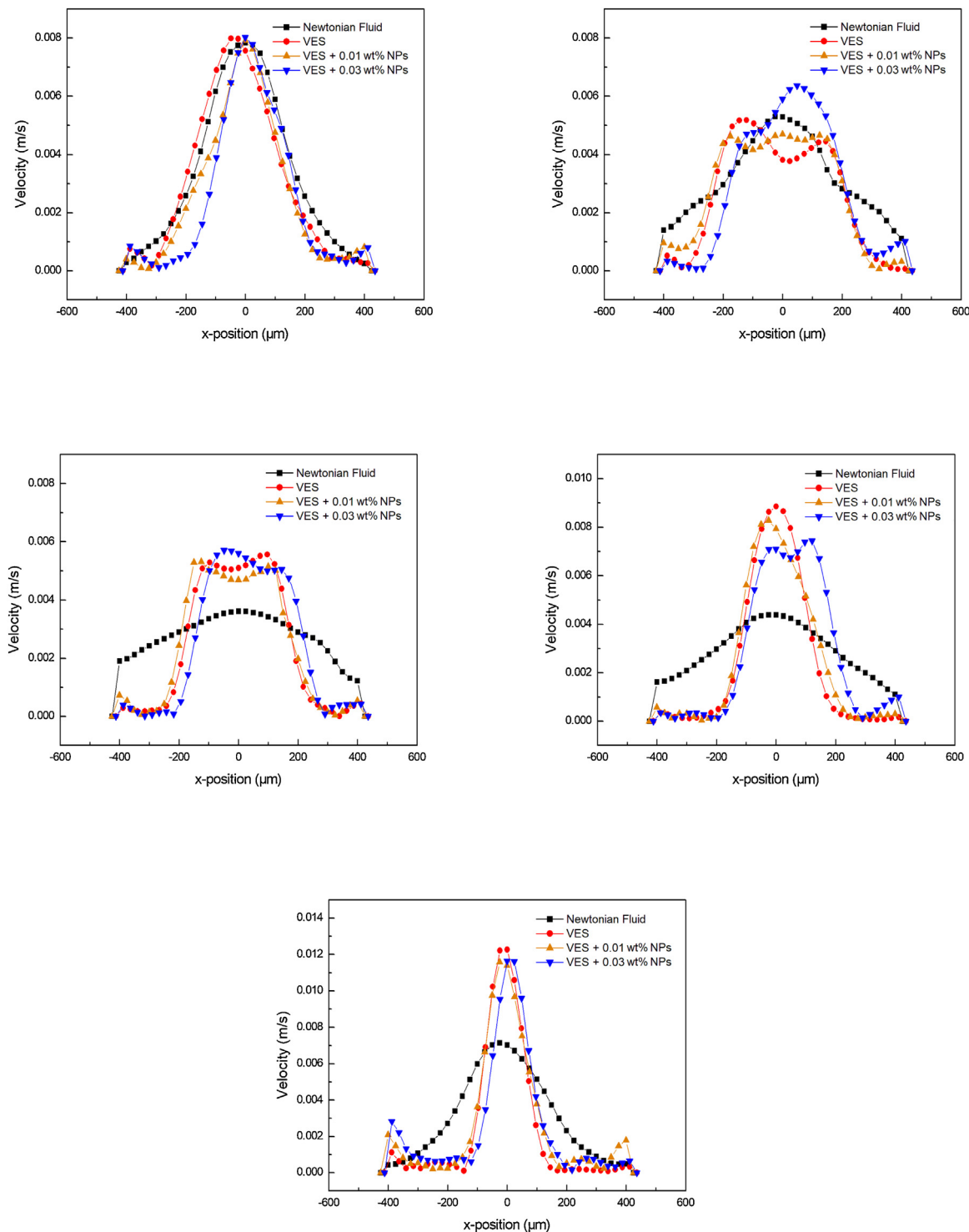
Fig. 9 – Schematic of the position where velocity data were extracted.

rates, even not working at all. There are barely flow patterns difference in Figs. 6, 7 and 8 at $Q = 5$ and 20 ml/h. Streamlines disperse from the centerlines to the boundary of the pore-throat structure at upstream and reunify near $y = -100 \mu\text{m}$ to $y = -50 \mu\text{m}$. The flow patterns and gathering points of streamlines are identical among the three systems, and the expected changing of flow characteristics fails to be obtained. Considering the velocity component of nanoparticle-enhanced VES fluids around the entrance of pore-throat structure at $Q = 5$ and 20 ml/h, the velocity component along y-direction increases rather than decreases the proportion of the actual velocity. It is surprisingly contrary to the current knowledge of the nanoparticle-enhanced effect on VES fluids.

For all VES fluids, they experience both shearing and stretching deformations while flowing through the

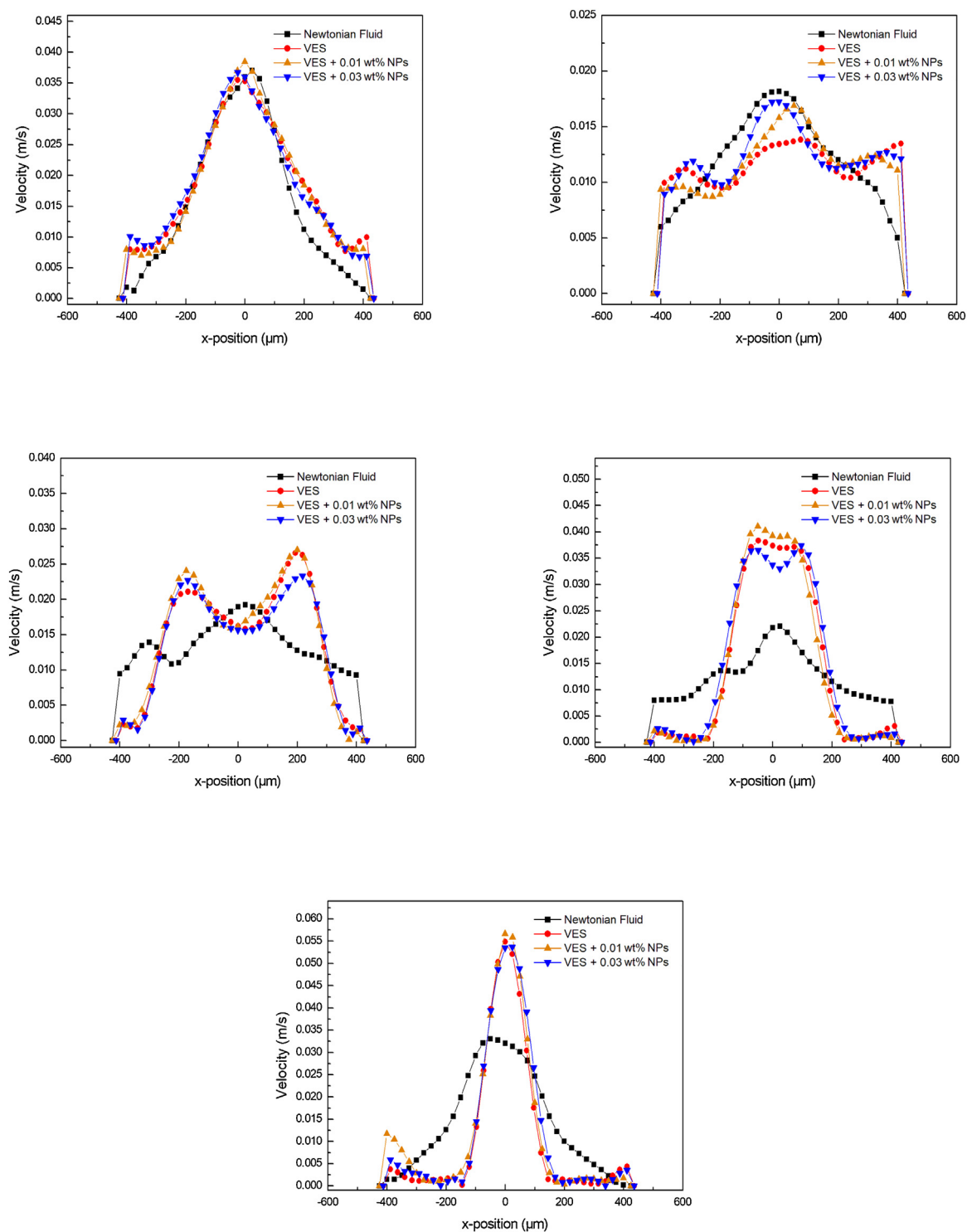
convergence–divergence or divergence–convergence structure. Breakage of entangled wormlike chains is under not only the shearing but also the stretching effect. Based on the rheological shear-viscosities of three VES fluids, their viscosities are similar with shear rates $\dot{\gamma}$ higher than 20 s^{-1} . Nanoparticle produces little effect on strengthening the

shear-viscosity of VES fluid at the high shear rate. In addition, since the contraction behavior of nanoparticle-enhanced VES fluids is not weakened, enhancement on extension-viscosity of VES fluid by nanoparticle is impracticable as well. In brief, the nanoparticle is unable to change the flow characteristics in the pore-throat structure.



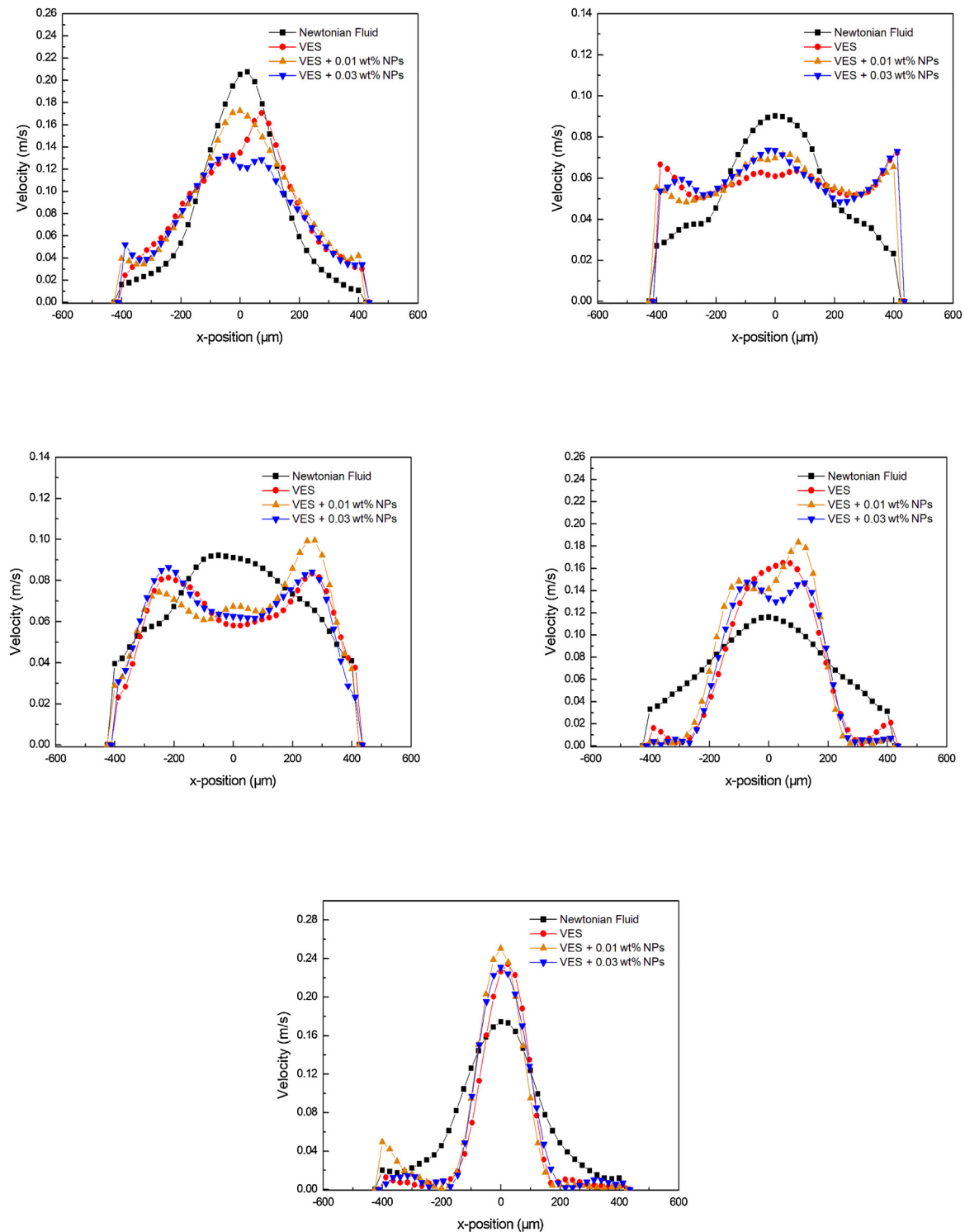
(a)

Fig. 10 – Velocity profiles in x direction of the Newtonian fluid, VES fluid, VES +0.01 wt% NPs fluid and VES +0.03 wt% NPs fluid flowing through the continuous pore-throat structure from $y = -340 \mu\text{m}$ to $340 \mu\text{m}$ with flow rate (a) $Q = 1 \text{ ml/h}$, (b) $Q = 5 \text{ ml/h}$ and (c) $Q = 20 \text{ ml/h}$. (For interpretation of the references to colour in the text, the reader is referred to the web version of this article.)



(b)

Fig. 10 – (Continued)



(c)

Fig. 10 – (Continued)

3.3. Velocity profiles in the x-direction

Five representative velocity lines along x-direction are listed to mark the line positions of different fluids at the same y-position of the pore-throat structure ($y = -340 \mu\text{m}$, $y = -170 \mu\text{m}$, $y = 0 \mu\text{m}$, $y = 170 \mu\text{m}$ and $y = 340 \mu\text{m}$) in Fig. 9. Fig. 10 illustrates the velocity profiles of four fluids investigated (including Newtonian fluid) to compare the distinction

of different systems at the same positions with the same flow rates.

Velocity profiles of three VES fluids (red for VES fluid, orange for VES + 0.01 wt% NPs and blue for VES + 0.03 wt% NPs) are highly matched with each other. Nanoparticle-enhanced VES fluids show no difference in velocity distribution. For low flow rate at $Q = 1 \text{ ml/h}$ in Fig. 10a, values at both sides in velocity lines of VES fluids are close to zero at $y = -170 \mu\text{m}$, $y = 0 \mu\text{m}$

and $y = 170\ \mu\text{m}$. Flows of VES fluids concentrate in the center areas in contrast to Newtonian fluid (black lines). For $Q = 5\ \text{ml/h}$ and $20\ \text{ml/h}$ in Fig. 10b and c, at $y = -340$, lines of four fluids are in coincidence. While at $y = -170\ \mu\text{m}$, both sides of VES fluids lines start to be higher than that of Newtonian fluid, indicating the reduction of structural viscosity. At $y = 0\ \mu\text{m}$, two velocity maximums emerge outside center area around $x = -250\ \mu\text{m}$ and $250\ \mu\text{m}$ for $Q = 5\ \text{ml/h}$, $x = -300\ \mu\text{m}$ and $300\ \mu\text{m}$ for $Q = 20\ \text{ml/h}$, respectively. However, unlike velocity calculated by Navier–Stokes equations (the velocity maximum is in the middle of velocity lines), the minimum value of VES fluid was found at the centerline. As for $y = 170\ \mu\text{m}$, two velocity maximums still exist while moving to center area at $x = -150\ \mu\text{m}$ and $150\ \mu\text{m}$ for $Q = 5\ \text{ml/h}$, $x = -180\ \mu\text{m}$ and $2000\ \mu\text{m}$ for $Q = 20\ \text{ml/h}$, respectively. Meanwhile, flow velocities at both sides decrease further, and streamlines regather to the center area.

Besides, at $y = 340\ \mu\text{m}$, the velocity profiles of all VES fluids show the same characteristics. Velocity lines of VES fluid without nanoparticle coincides with two nanoparticle-enhanced VES fluids, about $0.012\ \text{m/s}$, $0.058\ \text{m/s}$ and $0.23\ \text{m/s}$. The contractive behavior of streamlines is also observed as entering into the divergence–convergence structure at downstream as well. The velocities of Newtonian fluid are apparently lower than those of VES fluids in the middle, while higher at both sides. This contraction of viscoelastic fluid has been discussed in our previous work (Li et al., 2019).

Conducted from the results above, firstly, the nanoparticle is unable to enhance or change the viscoelasticity of VES fluid when flowing through convergence–divergence structure or divergence–convergence. Neither the breakage nor the recombination of wormlike micellar is barely affected by the nanoparticle. Secondly, although the increasing mechanism of rheological properties by nanoparticles has been proven before (Huang and Crews, 2008; Dai et al., 2017), it is excluded from the decreasing reason for the filtration loss. Finally, the filtration loss of nanoparticle-enhanced VES fluid is more likely to be controlled by the formation of nano-filter cake.

4. Conclusions

In conclusion, core experiments indicate that the filtration losses of VES fluid with no nanoparticles, VES +0.01 wt% NPs and VES +0.03 wt% fluids exhibit significant differences. Although the shear-viscosities of nanoparticle-enhanced VES fluids measured by the rheology test are strengthened while shearing rates $\dot{\gamma} < 10\ \text{s}^{-1}$, flow patterns in microchannel show different results with Deborah numbers rising from 1.52 to 73.2. The enhancement of nanoparticle on the structural strength of wormlike micellar in VES fluid is not significant at high flow rates ($5\ \text{ml/h}$ and $20\ \text{ml/h}$). Besides, the enhancement of extension-viscosity of VES fluid by nanoparticle is impracticable as well. Both the convergent and divergent flow patterns are barely changed among all VES solutions. Adding nanoparticles into fracturing fluids is able to reduce the filtration losses. However, this reducing effect is insignificant by the viscosity enhancement of nanoparticles.

Conflicts of interest

There are no conflicts of interest to declare.

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