Enhance The Properties of Cellulose Fracturing Fluids By Cellulose Nanocrystals

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Abstract

With inspiration from the enhancement of the viscoelasticity of cellulose nanocrystals (CNCs) on hydrogels, we paid attention to cellulose fracturing fluids prepared from the CNCs skeleton reinforced covalently cross-linked cellulose networks. In this paper, we hybridized CNCs with sodium CMC-Na to form nano cellulose fracturing fluids, characterized the performance of nano hybrid fracturing fluids and analyzed the nano-crystals enhancing fracturing fluid performance mechanism. It showed that the apparent viscosity, temperature tolerance, elastic modulus, and tensile strength of cellulose fracturing fluids were improved by CNCs. Compared with the blank cellulose fracturing fluids, the viscosity value of nano-cellulose fracturing fluids at 120 ℃ reached 195mPa·s, with an increase of 200 %, and the elastic modulus was 30Pa, with an increase of about 253 %. Furthermore, the microscopic analysis revealed that CNCs can play the role of nucleus and skeleton in the network structure of cellulose fracturing fluids and obviously enhanced the strength of the network structure. It is obvious to improve the performance of the fracturing fluids by adding CNCs into the cellulose fracturing fluids. This method provides a new idea for the development of fracturing fluids suitable for unconventional reservoirs.

Introduction

In recent years, with the advancement of exploration and development technology, more and more unconventional reservoirs have been explored and developed (Jia et al. 2012; Sun et al. 2019; Li et al. 2020). They are characterized by high temperature, tight and deep burial, so reservoir stimulation has become an indispensable and efficient development method (Lau et al. 2016; Yang et al. 2018; You et al. 2018; Dai et al. 2019). Currently, for the development of tight oil and gas, and other resource reservoirs, it is particularly important to improve the performance of fracturing fluids (Scanlon et al. 2015; Zhao et al. 2018). Conventional water-based fracturing fluids include guar gum fracturing fluids, cellulose fracturing fluids, and polymer fracturing fluids (Adewole and Muritala 2019). Among them, guar gum and polymers have some disadvantages such as relatively narrow supply, high residue content, and high cost (Barati and Liang 2014; Thombare et al. 2016; Sharma et al. 2018). Therefore, cellulose, which has the advantages of low cost, wide sources, and no pollution, has become the best choice for fracturing fluids thickeners for low-cost and efficient development of unconventional reservoirs (Yang et al. 2018; Gao et al. 2019).

Despite the excellent properties of cellulose, its industrial application is limited owing to its poor water solubility. Given the poor water solubility of traditional cellulose, previous researches focused on improving its water solubility by etherification (Sang and Xiao 2009; Chieng and Chen 2010; Hebeish et al. 2010; Sehaqui et al. 2016), esterification (Wang et al. 2018), graft copolymerization (Raus et al. 2011; Thakur et al. 2013). To ensure its good water solubility, a branched chain was introduced into the main chain, which breaks the length of the chain, so that the molecular chains are not entangled enough (Chang and Zhang 2011). Therefore, the thickening effect is not good, and the shear resistance and temperature resistance of the network structure are limited. As a result, cellulose fracturing fluids can not

meet the temperature resistance requirements of the unconventional reservoir at 120 ^oC using the modified methods mentioned above.

Some scholars innovatively added nanomaterials to viscoelastic fluids and found that adding nanomaterials can significantly enhance the strength of the network structure (Nettesheim et al. 2008; Helgeson et al. 2010). Nanoparticles with a high specific surface area can interact with more crosslinking agents to enhance the crosslinking effect between crosslinking agents and polymers. This effect not only improves the network density but also significantly reduces the amount of crosslinking agent, which ensures the good stability of the nanocomposites at high temperatures (Pal et al. 2015; Budnyak et al. 2018). In recent years, nanomaterials have been widely used in the field of oil and gas field stimulation (Lafitte et al. 2012; Lau et al. 2016; Villada et al. 2021). At present, nano Al_2O_3 (Savvashe et al. 2016), TiO_2 (Hurnaus and Plank 2015), ZrO_2 (Tana et al. 2017), and other materials have been added into the polymer to form a hydrogel with excellent mechanical properties. Fakoya and Shah (2018) added nano-SiO_2 to surfactant fracturing fluids and polymer fracturing fluids, which improved the rheological properties and temperature resistance of the fracturing fluids and changed the wettability of the rock. Crews and Huang (2008) added nanoparticles with a mass ratio of 0.12 wt.% to 0.18 wt.% to clean fracturing fluids with a surfactant molecule volume ratio of 2 wt.% and maintained an apparent viscosity of 300 mPa·s at 65°C and 100 s^{-1}. Regarding fluid loss prevention and sand carrying stability, the performance of nano-composite fracturing fluids is significantly improved. Luo Mingliang (2012). also used nano-modification of a fracturing fluids based on anionic surfactant fatty acid methyl ester sulfonate. Under the conditions of 70 °C and 170 s^{-1}, its apparent viscosity can reach 50 mPa·s.

Among many nanomaterials, we focus on cellulose nanocrystals(CNCs), which are highly crystalline rod-like nanomaterials isolated from cellulose. Compared with traditional nanomaterials, CNCs has the advantages of environmental friendly, wide source, renewable, mass production, and low cost, and also can be well matched with cellulose and its derivatives(Lu and Hsieh 2010; Moon et al. 2011; Liu et al. 2021). In addition, cellulose nanocrystals have excellent mechanical properties, such as high crystallinity (60%~90%), high elastic modulus (150 GPa), and high tensile strength (10 GPa) (Habibi et al. 2007; Iwamoto et al. 2009; Yang et al. 2021). Yang and co-workers (2013) covalently cross-linked cellulose nanocrystals and polyacrylamide to prepare two component composite hydrogels with excellent mechanical properties and recovery ability. By means of the indentation depth sensor, the contribution of CNCs skeleton to hardness and elasticity increased by 500% compared with the original PAAm hydrogel. Ahn and Song (2016) analyzed the viscoelastic characteristics of all cellulose nanocrystals and carboxymethyl cellulose (CMC-Na) suspensions and verified that cellulose nanocrystals can be well dissolved together, which provided a favorable theoretical foundation for cellulose nano fracturing fluids. However, there have been few studies on hydrogels formed by Cellulose fracturing fluids and CNCs. Inspired by the above, this paper innovatively mixed cellulose nanocrystals and cellulose to form hydrogel with high strength.

Herein, we hybridized CNCs with sodium CMC-Na to form cellulose nano fracturing fluids, analyzed the mechanism of cellulose nanocrystals and cellulose, studied the rheological properties, temperature resistance, and gel-breaking performance of nano-hybrid fracturing fluids. The present work provides a new approach to improve the properties of cellulose fracturing fluids via the use of CNCs as both cross-linker and nanofiller.

Experiments

Materials

CNCs, CMC-Na (≥ 96%), and glacial acetic acid (≥ 99%) were all supplied by Kelong Chemical Reagent Co., Ltd (Chengdu, China). Zirconium Butoxide (80wt%) and triethanolamine (99wt%) were purchased from Aladdin Industrial Corporation (China). All chemicals and solvents used in the experiments are analytical reagents and can be used without any purification.

Methods

Preparation of fracturing fluids

CNCs were added into 100mL ultrapure water with different concentrations (500 ppm, 800 ppm, 1000 ppm, 1200 ppm 1500 ppm), and then solutions were stirred in a water bath at 60℃ for 15 ~ 20 min to form a uniform dispersion solution. 0.4 wt.% CMC-Na added to the cellulose nanocrystalline solution and magnetically stirred (1000 rpm) for 2 h to obtain nanohybrid fracturing fluids base solution. After that, zirconium butanol and triethanolamine are mixed at a 1:1 ratio to form a complex cross-linking agent. The crosslinker was added to the nanohybrid fracturing fluids base solution to carry out a crosslinking reaction at room temperature and form a gel. The cellulose nanocrystalline and complex cross-linking agent concentration in prepared hydrogel varied from 500 to 1500 ppm w/w and 0.03 to 0.07 wt.%, respectively. The carboxymethyl cellulose fracturing fluids without cellulose nanocrystalline was prepared by adding 0.4 wt.% CMC-Na to ultrapure water and stirring for 2 h; then, 0.06 % cross-linker was added to the solution to form a gel.

Rheological measurements

The apparent viscosity, temperature resistance, and viscoelasticity of the cellulose nano fracturing fluids and blank fracturing fluids (without CNCs) were tested using a Haake Mars III rheometer (Thermo Scientific, Germany). A P39 rotor was used for the apparent viscosity test of all kind of fracturing fluids under 25°C continuous shear for 60 min at 170 s^{-1}. The temperature resistance of the fracturing fluids was determined at a shear rate of 170 s^{-1} and different temperatures (20°C ~ 120°C). A P35HAL rotor was used for the viscoelasticity test, which included strain scan, stress scan, frequency scan, and time scan.

Sand carrying performance

At room temperature, the basic fracturing fluids was prepared and mixed with proppant (30/50 Carbo ceramics, concentration 80kg/m3). After complete mixing, the pH regulator and crosslinking agent were added until the crosslinking was completed. Next, the sand mixture was poured into the static sand-carrying experimental flat plate device (600 mm×200 mm×10 mm) for static setting until the gas in the device was completely emptied. The distance between the proppant and the bottom was recorded every 5 minutes.

Microstructure Characterization

The dispersion and distribution of cellulose nanocrystals in fracturing fluids were characterized with a Quanta 450 environmental scanning electron microscope and Tecnai G2F20S-TWIN field emission transmission electron microscope (SEM, TEM, FEI Company of USA). Scanning electron microscopy experiments were carried out to observe the microstructure of the nano cellulose fracturing fluids at room temperature (25°C). The uniformly distributed hybrid fracturing fluids foundation was dropped on a copper mesh, and the microstructure of the cellulose nanocrystals in the fracturing fluids was observed by transmission electron microscopy at room temperature.

Fracturing fluids were investigated for their decomposition temperature with a thermogravimetric analyzer (TGA, LF DC-40MT, Mettler Toledo, Switzerland). The samples were heated from 40°C ~ 600°C at a rate of 20°C/min under nitrogen protection.

Results And Discussion

Apparent viscosity test

It is better to transport sand into fractures with higher viscosity fracturing fluids, which can be improved by adjusting the amount of crosslinking agent. The performance of the fracturing fluids was influenced by the cross-linking ratio, temperature, and concentration of CNCs.

The rheometer was utilized to test the apparent viscosity of fracturing fluids systems with different concentrations of cellulose nanocrystals and cross-linking ratios. As presented in Fig. 2, with the cross-linking ratio increased, the apparent viscosity value increased initially and then decreased, which showed the addition of proper concentrations of CNCs could significantly improve the viscosity of fracturing fluids. However, the performance of the fracturing fluids was not significantly improved when the concentration of nanomaterials increased. This was probably due to the nano effect which causes a small amount of CNCs to aggregate into agglomerates and leads to poor dispersion of CNCs. Under the condition of low crosslinking agent concentration, the cross-linking of the cellulose chain with zirconium crosslinker was insufficient and the network structure that formed was weak. On the other hand, the fracturing fluids gel would be over-crosslinked under the condition of higher cross-linking agent concentration and it would show strong brittleness and result in a decrease in the viscosity of the fracturing fluids.

It was shown in Fig. 2 that when the concentration of CNCs was 1000ppm and the cross-linking ratio was 0.04 wt.%, the apparent viscosity reached the maximum of 683.3 mPa·s. Moreover, the optimal crosslinking ratio of the blank cellulose fracturing fluids was 0.06 wt.% reaching the viscosity of 550 mPa·s. It indicated that CNCs played an important role of "crosslinker" in fracturing fluids. The metal crosslinker groups were connected to its surface through chemical bonds and then cross-linking with CMC-Na molecules, resulting in the decrease of the amount of crosslinker.

Elastic modulus test

As presented in Fig. 3a-c, with the increase of CNCs, the elastic modulus G' became more stable. The yield stress and elastic modulus increased at the beginning and then decreased; the tanδ value was equal to 1. The critical point was also moved to the right first, corresponding to a higher critical stress value. After that it moved to the left, corresponding to a lower critical stress value. These experimental results showed that a proper amount of CNCs can improve the elastic (solid) characteristics of the sample and reduce the viscosity (fluid) characteristics of the sample. This indicated that CNCs can enhance the complexity of the molecular chain conformation of cellulose in aqueous solution and make the microstructure transition from a simple network structure to a complex spatial network structure.

The results of the frequency scan were illustrated in fig 3 that with the addition of CNCs concentration, the initial value of the elastic modulus G' of the cellulose fracturing fluids increased and the frequency response was more stable and the linear viscoelastic interval was longer. When the number of CNCs increased, the changing trend of G' and the linear viscoelastic zone was to increase and extend firstly, which showed the highest and longest when the concentration of CNCs reach 1000ppm. After that, G' and linear viscoelastic zone began to decrease and be shorten. The better elastic properties of adding CNCs also reflected that the cellulose fracturing fluids with an appropriate amount of CNCs can have a stronger spatial network structure in water.

After determining the yield stress and the linear viscoelastic region, the sample was scanned for 300 seconds (the scanning stress value is 0.6 σ, and the scanning frequency is the midpoint of the linear viscoelastic region) in order to assess the elastic modulus. According to the time scanning results, the average G 'size of the 6 samples in the linear viscoelastic interval was 1000 ppm > 800 ppm > 500 ppm > 120 0 ppm > 0 > 1500 ppm. It indicated that the elastic modulus increased at the beginning and then decreased with the increase of CNCs concentration. It indicated that CNCs can improve the elastic performance of cellulose fracturing fluids, make the space network structure more stable, improve the shear resistance and the sand-carrying performance of cellulose fracturing fluids. In addition, it also proved that CNCs can act as a "cross-linking agent". Excessive addition of CNCs would lead to over-crosslinking of cellulose, resulting in unstable spatial network structure and indirectly impaired sand-carrying capacity.

The cellulose nano fracturing fluids at a concentration of 1000 ppm CNCs and a crosslinking rate of 0.04 wt.% generally showed better elastic characteristics, which was consistent with the previous conclusions of apparent viscosity (reference). Compared with the blank sample, the modulus G’ of stress scan,

frequency scan, and time scan increased by 175%, 200%, and 353% respectively under the conditions of 1000ppm of CNC and 0.04 wt.% cross-linking ratio.

Static particle settling test

The sand-carrying performance of fracturing fluids is related to the successful delivery of proppant to the target zone and the settlement distribution of proppant during the hydraulic fracturing, thus affecting the fracture geometry, conductivity, and the final productivity of the stimulated well. Accordingly the sand-carrying performance of fracturing fluids is an important point in performance evaluation. Therefore, the sedimentation rate of proppant of cellulose nano fracturing fluids with different CNCs dosage, guar gum fracturing fluids, and cellulose fracturing fluids were analyzed.

It was shown in Fig.4a-b that the addition of CNCs can significantly reduce the sedimentation rate of the proppant. In addition, with the increase in the amount of CNCs, the sedimentation rate decreased and then slightly increased, reaching a peak at 1000 ppm. This was mainly because the addition of CNCs increases the viscoelasticity of the cellulose fracturing fluids, thereby improving the sand carrying performance of the cellulose fracturing fluids correspondingly. With different amounts of CNCs, the viscoelasticity was different. The sedimentation rate decrease was also different, which was consistent with the previous viscoelasticity test results (the elastic modulus first increased and then decreased with the concentration of CNCs and reached the maximum at 1000ppm).

The proppant sedimentation process and sedimentation velocity of different fracturing fluids were showed in Fig. 4c-d. It suggested that the cellulose nano fracturing fluids was superior to guar fracturing fluids and cellulose fracturing fluids in sand carrying performance. It demonstrated that the cellulose nano fracturing fluids can transport proppant easily and is better to be applied in oilfield.

Temperature resistance test

The addition of CNCs had a remarkable influence on the temperature resistance properties of fracturing fluids. The relationship between the viscosity and temperature of cellulose nano hybrid fracturing fluids, guar gum fracturing fluids, and cellulose fracturing fluids at a shear rate of 170s-1 and 100 °C were showed in Fig. 5. It revealed that the addition of CNCs can greatly increase the viscosity of the cellulose fracturing fluids, indicating that CNCs can combine the hydroxyl groups of cellulose through hydrogen bonding and led to significantly increase the viscosity of the fracturing fluids and the stability of the network structure. With the increase of temperature, the viscosity of the three fracturing fluids gradually decreased, and the trend of cellulose nano fracturing fluids was obviously delayed. The viscosity of nano cellulose fracturing fluids was still 195 mPa·s at 120min, which means that CNCs can effectively increase the thermal stability of fracturing fluids. As shown in Fig. 6, compared with the other two fracturing fluids, cellulose nano fracturing fluids showed excellent temperature resistance and high viscosity at 120 °C.

Interaction mechanism between CNCs and CMC-Na

(1)TGA

The principle of thermogravimetric analysis is a method of measuring the relationship between the mass of a substance and temperature or time by controlling the temperature. From the above tests, we can find that nanocrystals improve the performance of cellulose fracturing fluids and thermogravimetric analysis verifies that cellulose nanocrystals improve the heat resistance of cellulose molecular chains.

It indicated from the TGA curve in Fig. 7 that the cellulose fracturing fluids and the cellulose nano fracturing fluids had similar processes throughout the process and there were two noticeable weight loss processes. 40℃~100℃ was the process of free water evaporation, 260℃~350℃ was the process of cellulose molecule decomposition, the decomposition temperature was 279℃ and 314℃, respectively, an increase of 12.54%. TGA proved that CNCs and CMC-Na through hydrogen bonding can help improve the temperature resistance of cellulose.

(2)SEM

The morphology of different fracturing fluids was observed using Quanta 450 Environmental Scanning Electron Microscope. It can be seen from Fig. 8a-c that the CMC-Na molecular chains were arranged linearly in the solution. From the SEM image of CNCs (Fig. 8d-f), CNCs were disorderly and scattered. Compared with pure CMC-Na, the addition of CNCs can make the sequence of CMC-Na molecular chains more compact and regular, shorten the distance between CMC-Na molecular chains, and thus increase the strength of CMC-Na molecular chains (Fig.8g-i). From Fig. 8j-l, the cellulose fracturing fluids showed a complex spatial network structure, but there was a local phenomenon of structural instability. By comparing Fig. 8j-l, it was found when CNCs has existed in the system, the spatial network structure of cellulose fracturing fluids was more substantial, with no hollow-out phenomenon and a more flat surface, which would enhance the mechanical characteristics of the spatial network structure of cellulose fracturing fluids.

(3)TEM

As presented in Fig. 9, CNCs played the role of nucleus and skeleton in the network structure of cellulose fracturing fluids. CNCs were prepared by hydrolysis of cellulose with sulfuric acid to remove the amorphous structure. The surface of the nanocrystals would have sulfuric acid group, so the surface of the nanocrystals would be negatively charged. Under the electrostatic repulsion action, the nanocrystals can be well dispersed in water. Because the nanocrystals and the nanocrystals had the same source, it was easy to mix them. With hydrogen bonding, the hydroxyl of the nanocrystals binds to the carboxyl of the CMC-Na molecular chain and the nanocrystals were completely surrounded by the cellulose molecular chain (Fig. 9). When the system was subjected to external shear, the rigidity and large surface area of the nanocrystals increased the probability of contact between cellulose molecular chains and nanocrystals. That resulted in the formation of hydrogen bonds soon after the destruction of the grid structure in the system, which keeps the grid stable. Therefore, the nanocrystals hybrid fracturing fluids showed high rheological characteristics.

Conclusion

In summary, the cellulose nano fracturing fluids with high strength of network structures have been successfully fabricated by CNCs and CNC-Na. Cellulose nanocrystals can be used as the core and skeleton of crosslinking in the network structure of cellulose fracturing fluid. CNCs increased the viscosity of fracturing fluids from 65 mPa·s to 195 mPa·s at a shear rate of $170 s^{-1}$ and 100 °C. The static sand carrying capacity increased significantly, and the proppant settling rate decreased by 40%. This work offers a new method for improving the cellulose fracturing fluids properties, which is expected to be extensively applied in the development of unconventional reservoirs.

Declarations

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

Compliance with Ethical Standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethical Statement Informed consent was obtained from all individual participants included in this study.

The manuscript is not currently being considered for published in another journal. This manuscript has not been published in whole or in part elsewhere.

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Figures

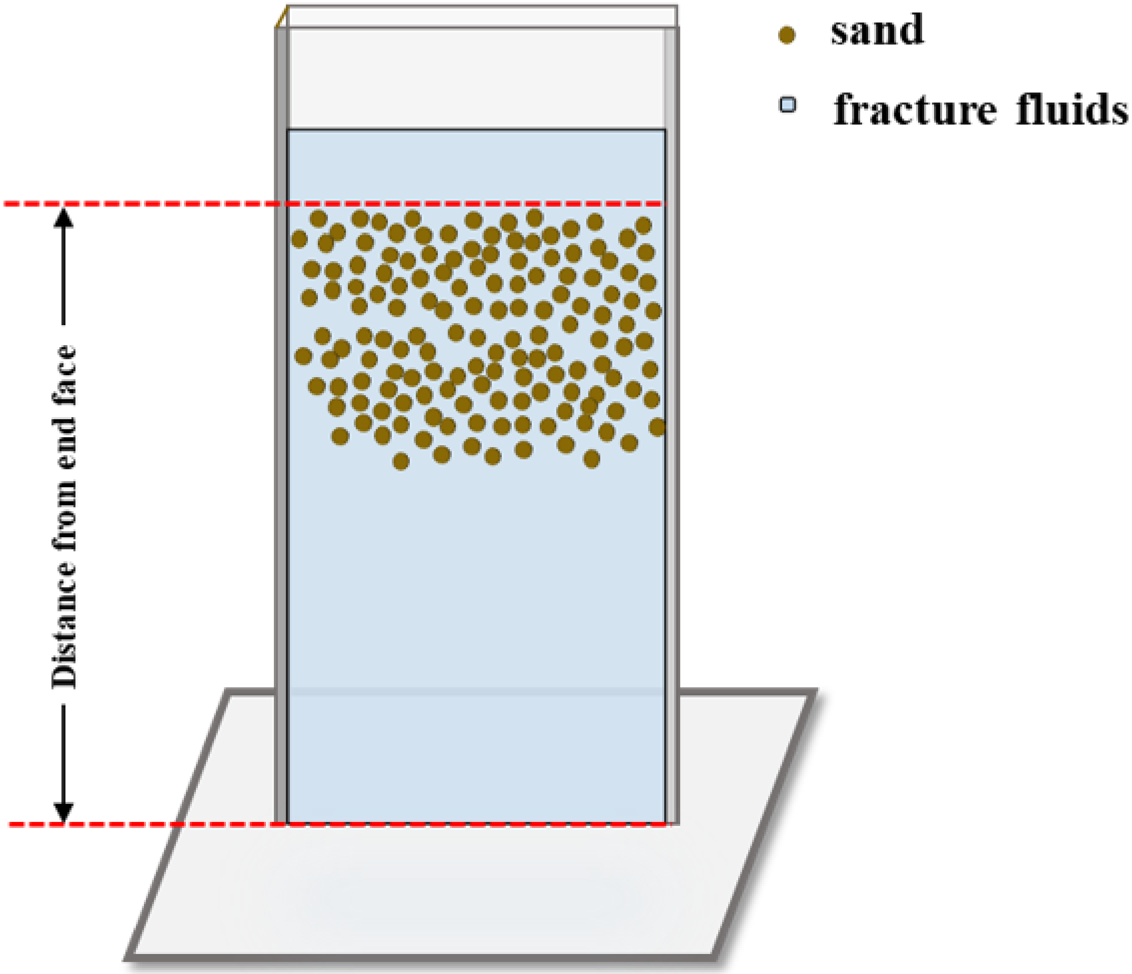

Figure 1

Static sand-carrying experimental flat plate device.

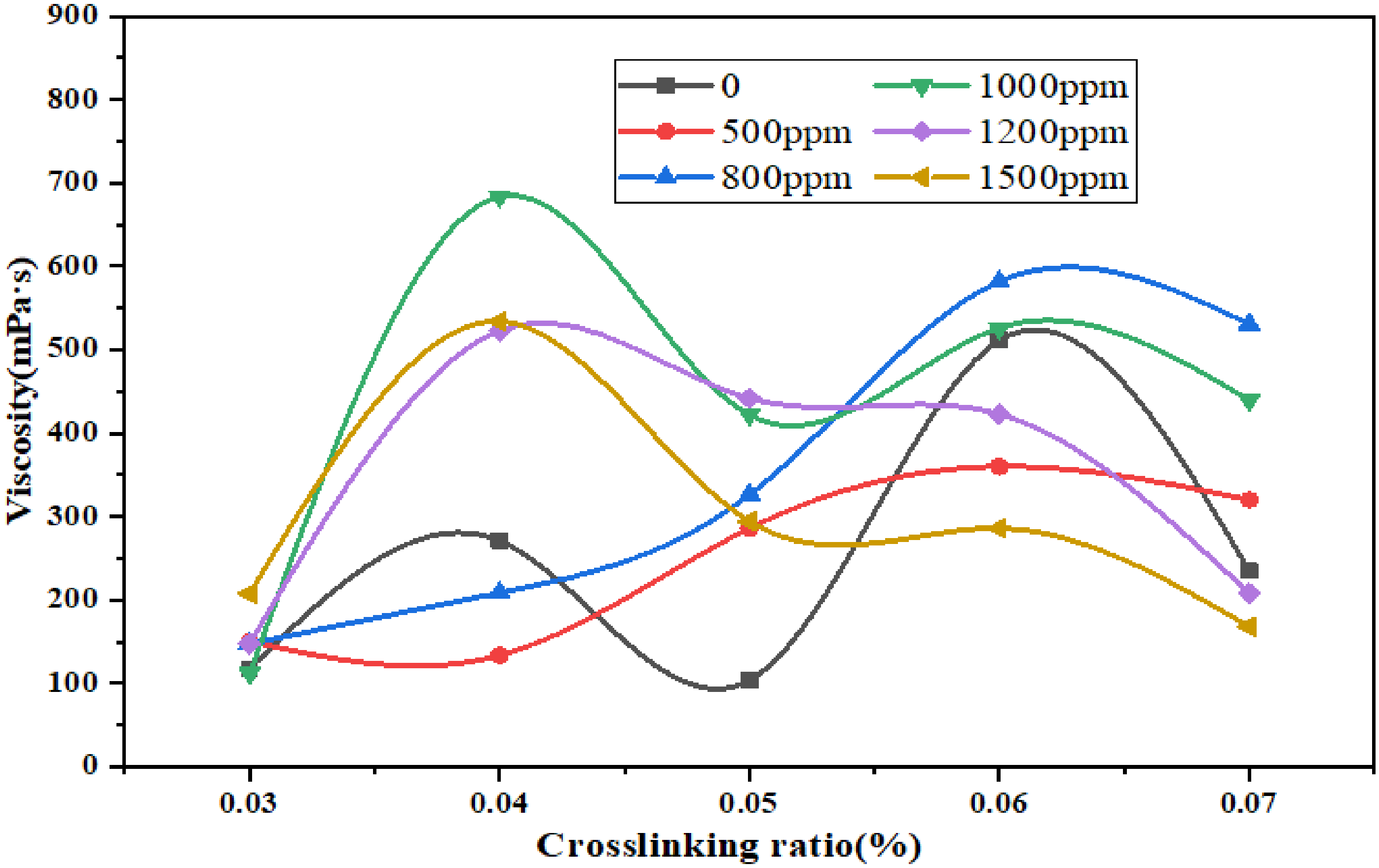

Figure 2

Relationship between viscosity and different concentrations of CNCs and crosslinking ratio.

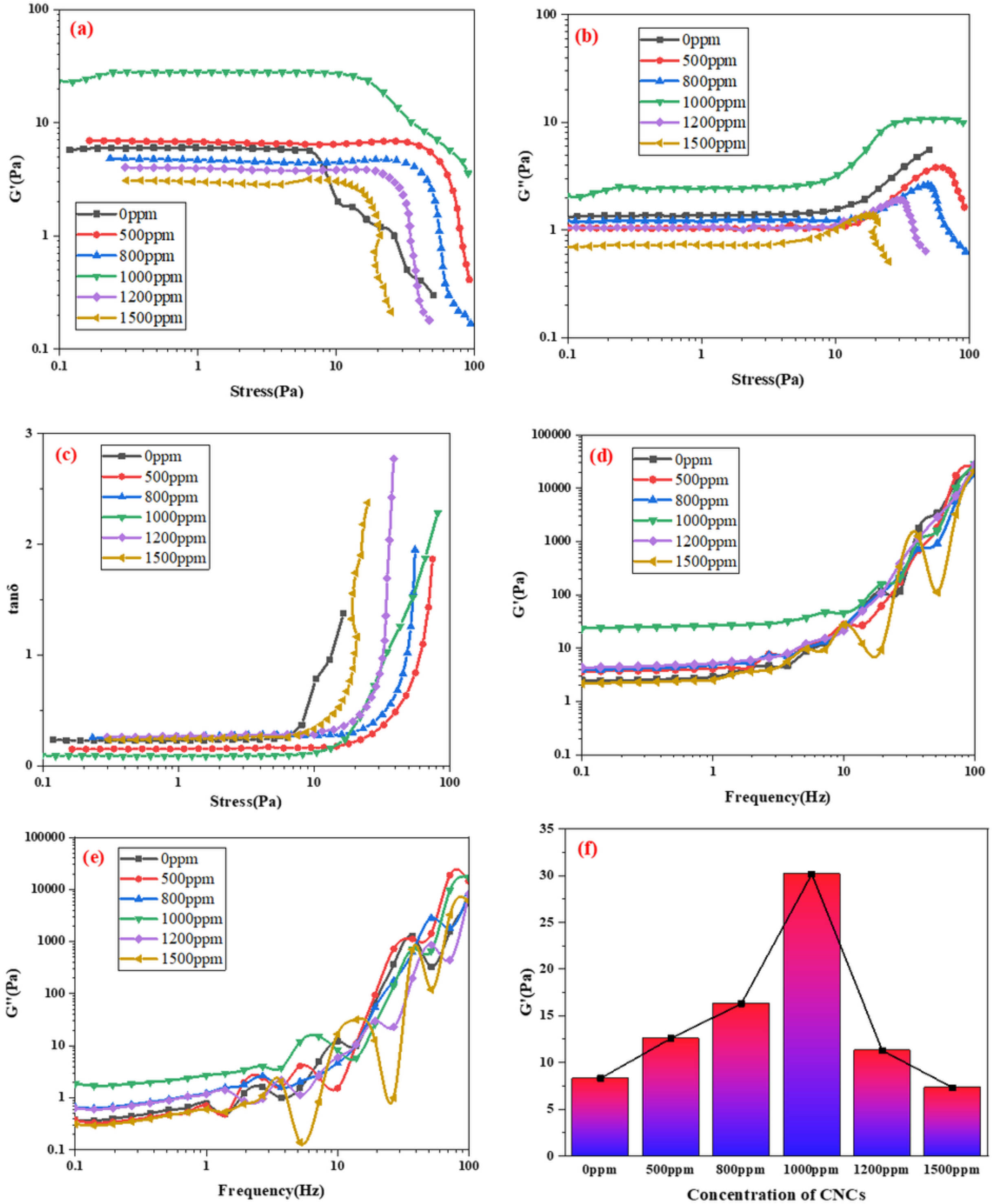

Figure 3

Viscoelastic test results of various concentrations of CNCs (stress scan (a, b, c), frequency scan (d, e), time scan (f)).

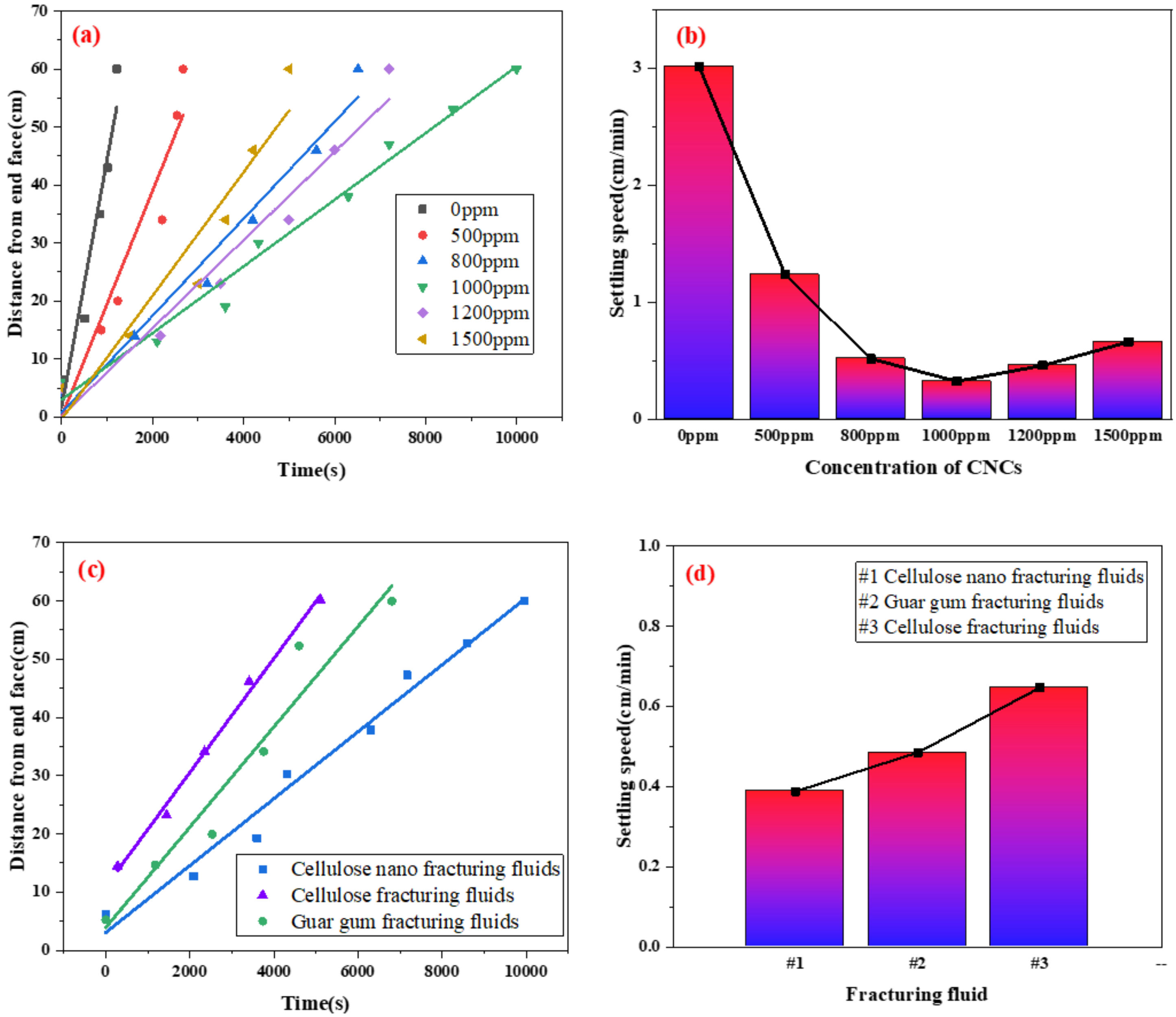

Figure 4

(a) Distance from the end face varies with the concentration of CNCs, (b) Settling speed for various concentrations of CNCs, (c) Distance from the end face varies with different fracturing fluids, and (b) Settling speed for various fracturing fluids.

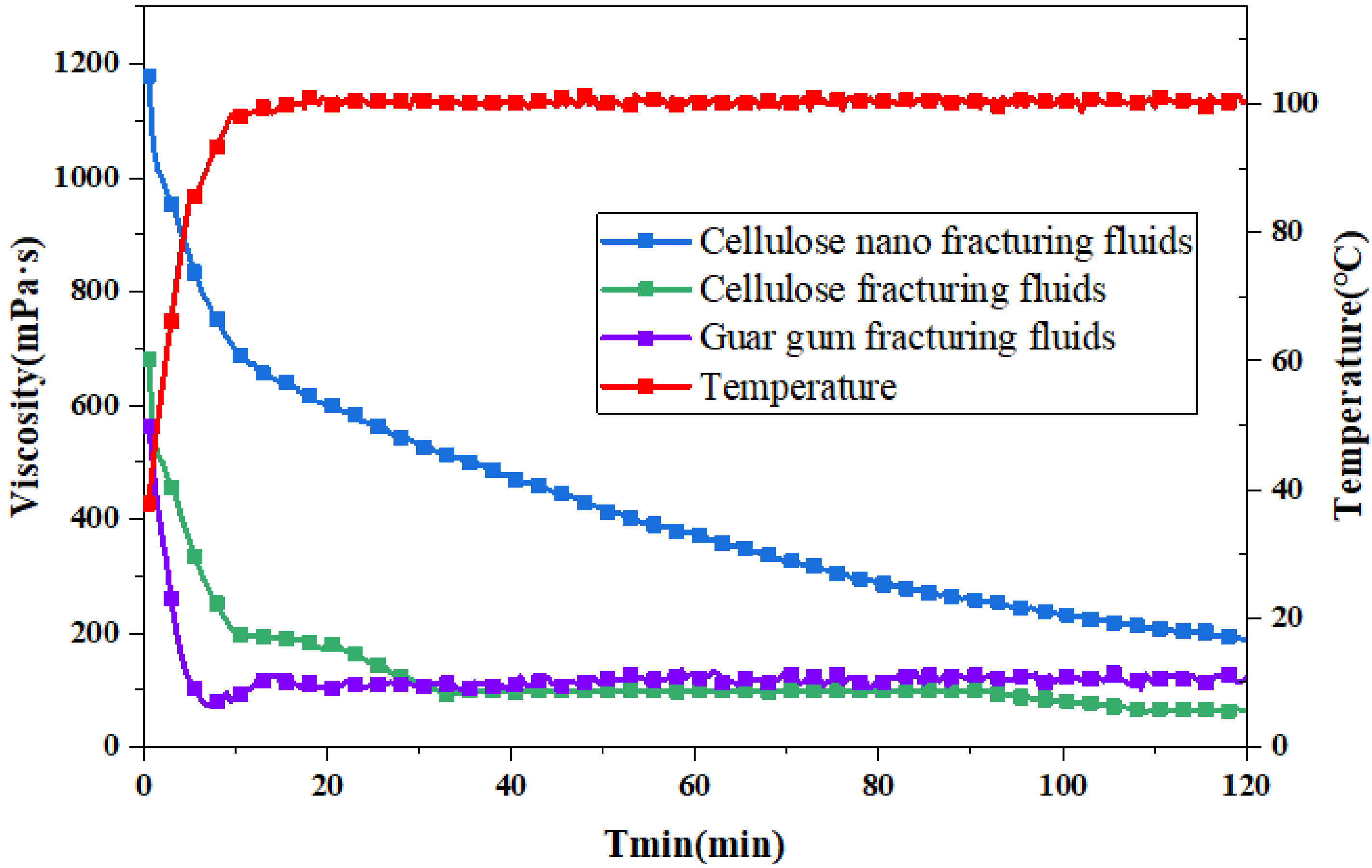

Figure 5

Relationship between the shear time and viscosity of fracturing fluids.

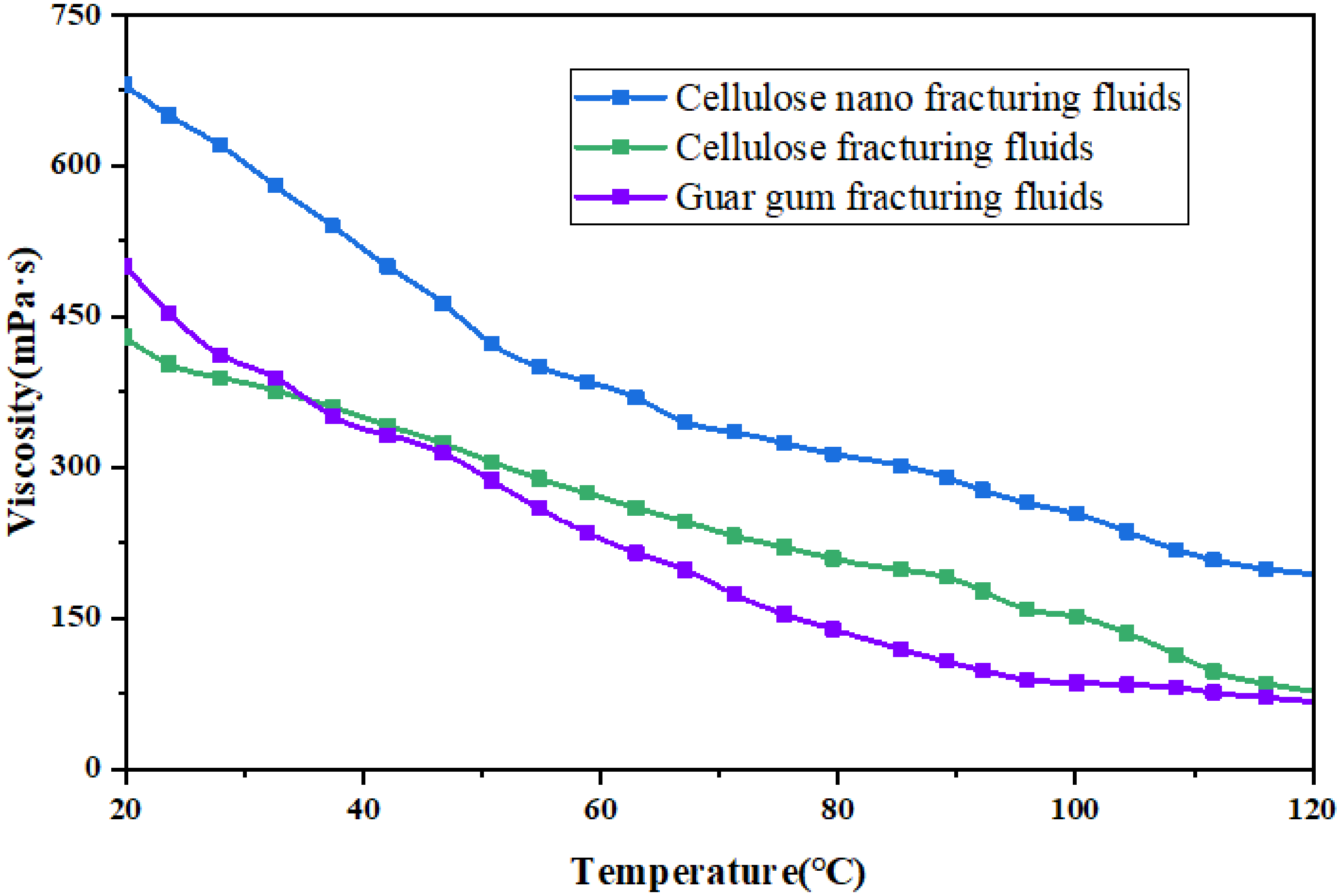

Figure 6

Relationship between the viscosity of fracturing fluids and temperature.

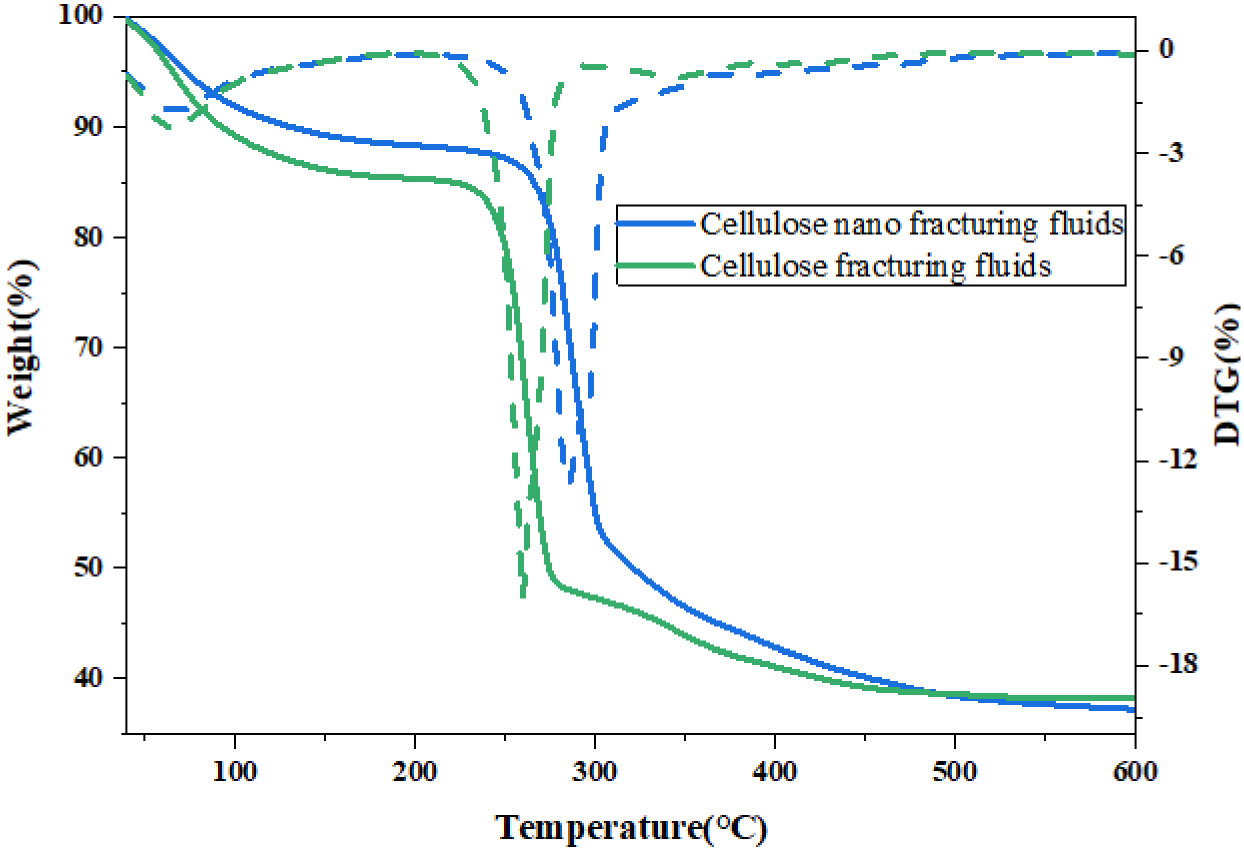

Figure 7

Thermal gravimetric curve of fracturing fluids.

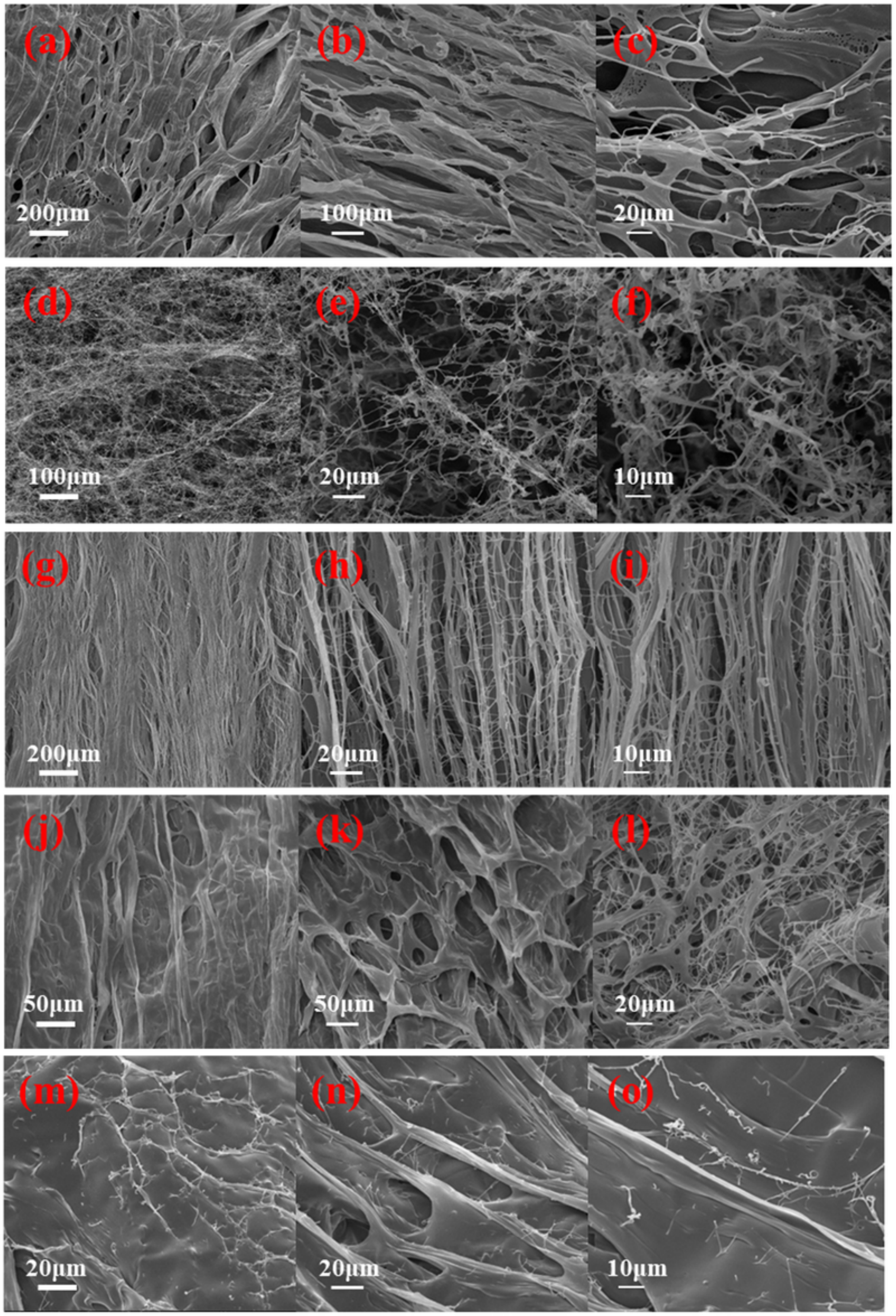

Figure 8

SEM images of (a)-(c) 0.4%CMC-Na, (d)-(f) 1000ppmCNCs, (g)-(i) 0.4%CMC-Na+1000ppmCNCs, (j)-(l) cellulose fracturing, (m)-(o) cellulose nano fracturing.

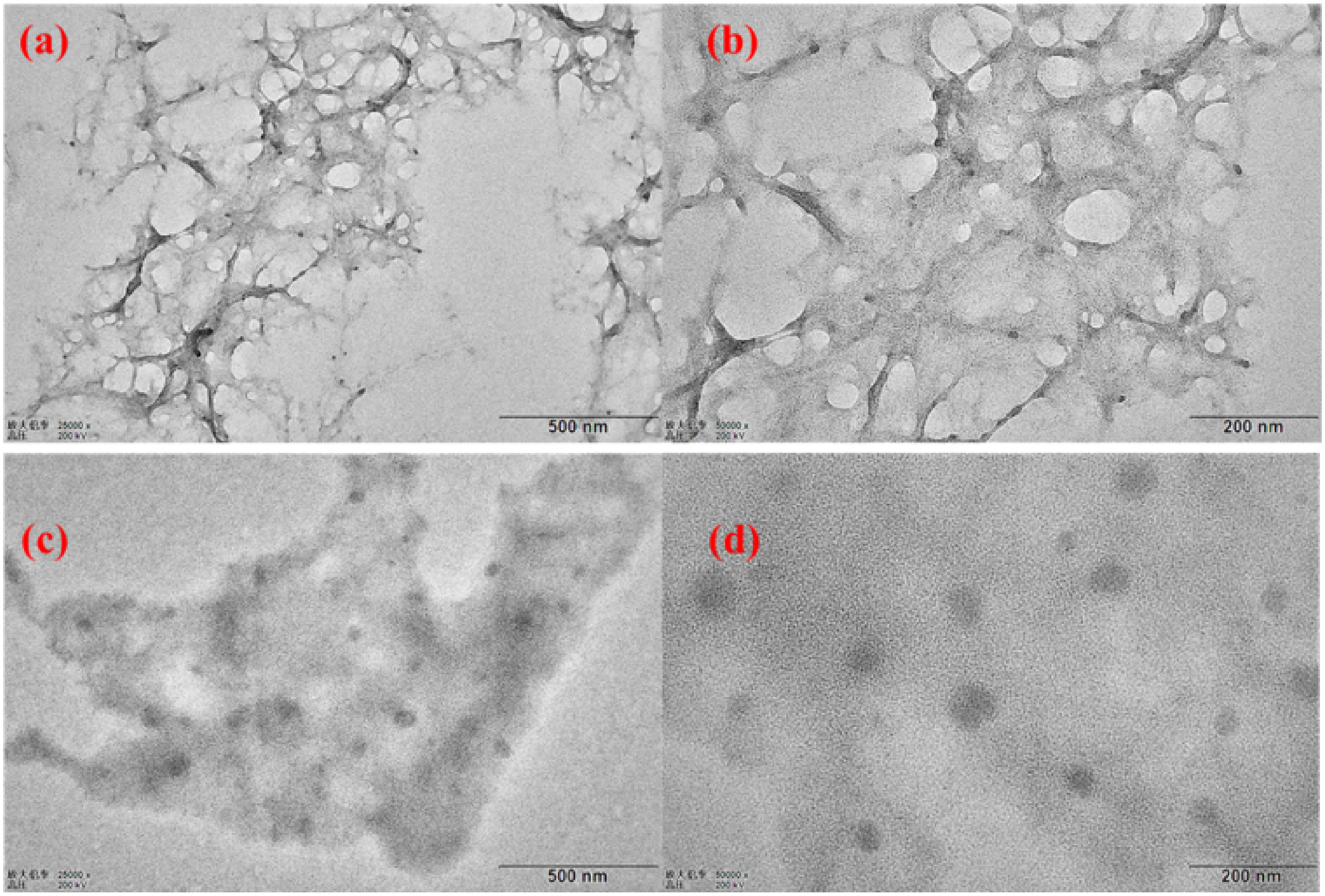

Figure 9

TEM image of the cellulose nano fracturing fluids (a-b) and the cellulose fracturing fluid (c-d).

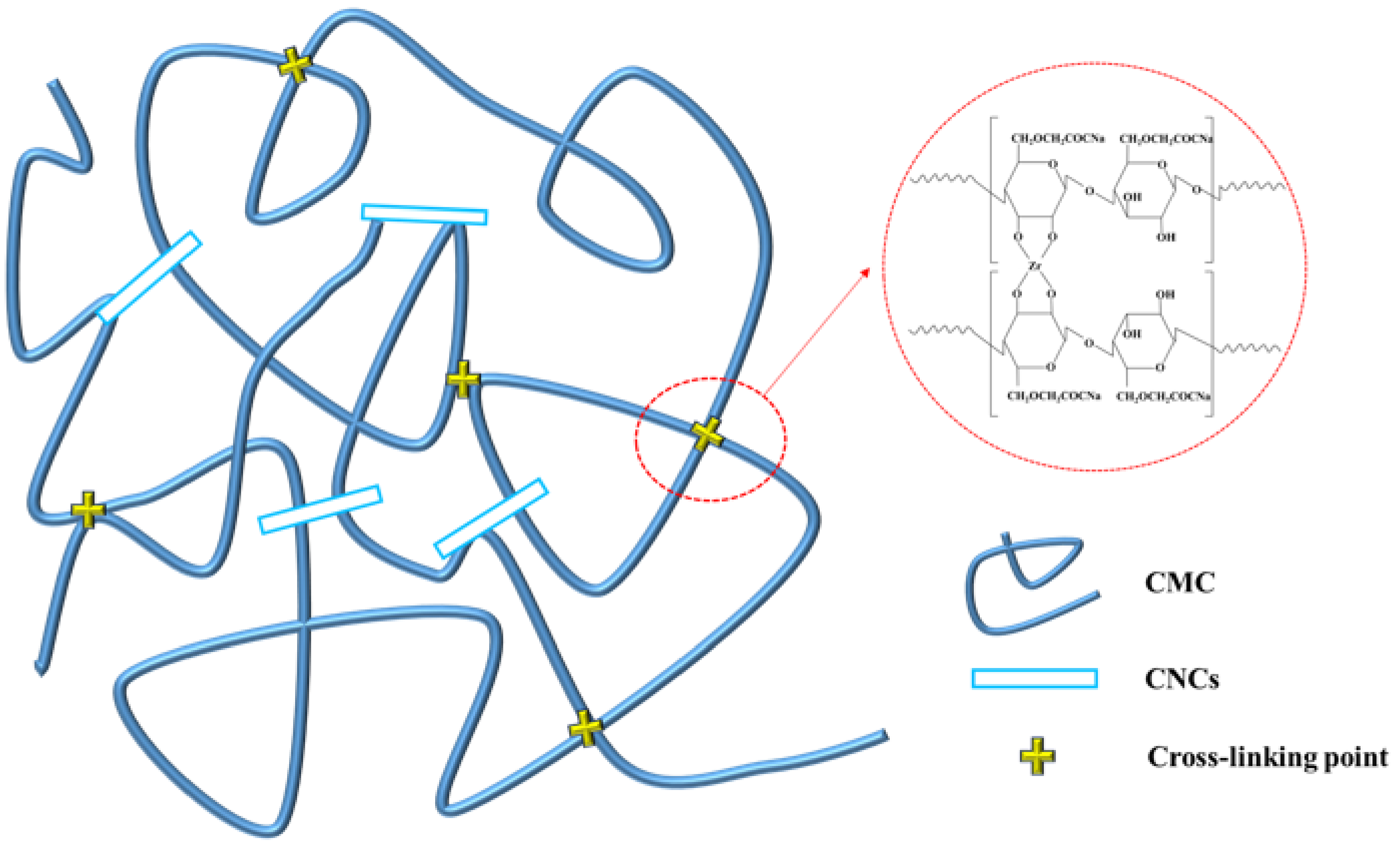

Figure 10

Schematic diagram of internal interaction of cellulose nano fracturing fluids.