

Synthesis and property evaluation of a salt- and alkali-resistant star-polymer

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Abstract: A salt- and alkali-resistant star-polymer was made using graft β -CD functional monomer and acrylamide monomer employing copolymerization and post-hydrolysis methods. The optimum parameters: azobisisobutyronitrile with a concentration of 50 mg/L, a mass fraction of acrylamide 25%, a graft β -CD functional monomer mass fraction of 1.5%, initiating temperature 5 °C, hydrolysis temperature 90 °C and hydrolysis time of 3 hours. The atomic force microscope and infrared spectroscopy morphology characterization results show that: the star-polymer S07313 contains the star-nucleus of the graft β -CD functional monomer. The molecular weight of the star-polymer S07313 is 24.5 million, while other basic physical and chemical properties can meet the technical requirements of the application of such a polymer in oil fields. With the addition of NaOH as a mass fraction of 1.0% in the Daqing simulated brine, the apparent viscosities of the star-polymer S07313, HPAM3000 and MO4000 are 42.40 mPa·s, 29.20 mPa·s and 18.00 mPa·s. The star-polymer S07313 and surface active agents Alkylbenzene Sulfonate have an excellent compatibility.

Key words: star-polymer; graft β -CD functional monomer; salt-resistance; alkali-resistance

Introduction

There are many temperature-resistant and salt-resistant polymers developed for tertiary oil recovery in China and abroad^[1–9]. They mainly include super-high molecular weight polyacrylamide, zwitterionic polymer, temperature-resistant and salt-resistant copolymers, hydrophobically associating polymers, multi-composite polymer, comb-polymer and star-polymer, however their alkali-resistant ability should be improved in further. Through analyzing and studying the salt- and alkali-resistant behaviour, it is considered that star-polymer has the most appropriate characteristics for future application. The study of salt- and strong alkali-resistant star-polymers suitable for composite flooding may provide theoretical accordance and technology support for enhancing oil recovery by composite flooding.

Star-polymer^[10–20] is made of one star nucleus and multiple super polymer chains, which is water-soluble with strong salt- and alkali-resistance. In this study, a salt- and alkali-resistant star-polymer was made by graft β -CD functional monomer and acrylamide monomer copolymerization and post-hydrolysis. The effects of some parameters on the copolymerization between a graft β -CD functional monomer and acryla-

mid, such as a multi-level complex initiation system, initiation temperature, mass fraction of acrylamide, mass fraction of graft β -CD functional monomer, mass concentration of azobisisobutyronitrile, hydrolysis temperature and hydrolysis time were also evaluated.

The structure of star-polymer was observed by atomic force microscopy and characterized by Infrared spectrum; as well the solution properties such as the basic physical chemistry, salt-resistance, alkali-resistance and the compatibility of the star-polymer were also evaluated.

1 Experiment

1.1 Main raw materials composing star-polymer

The main materials include: alkylbenzene sulfonate (Industrial), acrylamide monomer (CR), graft β -CD functional monomer (in-house), azobisisobutyronitrile, ammonium persulfate, NaOH, NaCl, sodium, KCl, MgCl₂, CaCl₂ and NaHCO₃, all of which are AR grade.

1.2 Synthesis method

The synthetic steps for star-polymer are as follows (1) Weigh the acrylamide monomer and graft β -CD functional

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monomer and put into a polymerization bottle. Take azobisisobutyronitrile by a pipette to the polymerization bottle. Add distilled water to the desired concentration; (2) The polymerization bottle was placed in a constant-temperature water bath, introducing nitrogen to remove oxygen for 25 min, then adding compound initiator while continuing to remove oxygen for a further 5 min. Stop the nitrogen, cover with a lid and take out the bottle, then conduct adiabatic polymerization; (3) after polymerization has reached the peak temperature, cure for 1 h, then take out the gel; (4) Weigh the gel, granulate and put into a hydrolysis bottle, adding an appropriate amount of hydrolysis agent and cosolvent. Put the hydrolysis bottle into a thermostatic bath and conduct the hydrolysis step for the required time; (5) Take out the finished gel particles, dry, crush and sieve them to obtain the white powdery star-polymer material.

1.3 Instrument

Main instruments: Intrinsic viscosimeter, electric mixer, electronic balance, NEXUS-470 infrared spectrometer, CSPM4000 Atomic Force Microscope produced by Primitivo Nano Instruments Company, temperature-control magnetic stirrer, DF-2002B electric heat oven, electric constant temperature water bath and glassware.

1.4 Determination method

Infrared spectroscopy measurement: the purified samples of star-polymer S07313 were crushed, made into a potassium bromide tablet, analyzed by the NEXUS-470 Fourier transform infrared spectrometer, recording the infrared spectra in the range of 400–4 000 cm^{-1} .

The surface morphology was observed by CSPM4000 atomic force microscopy: deionized water was used to prepare a sample solution with a polymer concentration of 1.00 mg/L. The solution was then vibrated and mixed by ultrasonic stirrer, adding one drop of the mixture (about 0.175 g) to a clean mica plate with diameter of 12.00 mm. The material was then spread and air dried naturally and an Atomic Force Microscope CSPM4000 was used to observe the morphology of star-polymer S07313 samples.

Intrinsic viscosity, molecular weight, filtration factor, apparent viscosity, water insolubility, dissolution speed and so on were all determined by oil and gas industry standards (SY/5862-93).

2 Results and discussions

2.1 Synthesis of star-polymer

In addition to investigated factors, the basic synthesis parameters were determined to be as follows: Azobisisobutyronitrile concentration of 50 mg/L, a mass fraction of 25% Acrylamide, a mass fraction of 1.5% graft β -CD functional monomer, an initiation temperature of 5 $^{\circ}\text{C}$, an hydrolysis temperature of 90 $^{\circ}\text{C}$ and hydrolysis time of 3.0 h was used.

2.1.1 Study on highly effective multi-level composite initiation system

According to the decomposition activation energy of the initiation system, the favorable decomposition temperature of the initiation system can be calculated. If the initiation system has a lower favorable decomposition temperature, the activation energy will also be lower. Usually single redox systems, such as a persulfate system, organic peroxide system, multi-electron transfer redox system or metal ion system^[3–5] have higher decomposition activation energies, therefore cannot initiate the copolymerization between the graft β -CD functional monomer and acrylamide when the temperature is lower than 20 $^{\circ}\text{C}$.

A multi-level complex initiation system (composed of an inorganic redox system, organic redox system and azobisisobutyronitrile) can be used to induce the polymerization between the graft β -CD functional monomer and acrylamide to prepare ultra-high molecular weight star-polymers (Table 1). The highly efficient multi-level complex initiation system used in this study has higher initiation activity, such that it can still initiate or even reduce the initial polymerization temperature to 0 $^{\circ}\text{C}$. The star-polymer produced by using a highly efficient multi-level complex initiation system has high molecular weight and good water solubility.

2.1.2 Effect of temperature

Figure 1 shows that when temperature is relatively higher or lower, the molecular weight of star-polymer will decrease in either scenario. Theoretically, low temperatures can increase the molecular weight of star-polymer, but the induction time is also prolonged, the initiator decomposition rate decreased, the polymerization rate slowed down and polymerization period prolonged, all of which are unfavorable for the control of production and may result in incomplete reactions. When temperature is relatively high, it will accelerate the

Table 1 Comparison of physical-chemical properties for star-polymers synthesized by different initiation systems

Initiation system	Initiation temperature/ $^{\circ}\text{C}$	Intrinsic viscosity/(dL g^{-1})	Molecular weight / 10^4	Apparent viscosity / ($\text{mPa}\cdot\text{s}$)	Water insoluble / %
Ammonium persulfate	50	16.58	1 100	24.70	0.08
Ammonium persulfate - sodium bisulfite system	25	20.65	1 535	33.60	0.10
Multi-level complex initiation system	5	28.12	2 450	63.90	0.18
Multi-level complex initiation system	0	27.20	2 330	60.50	0.19

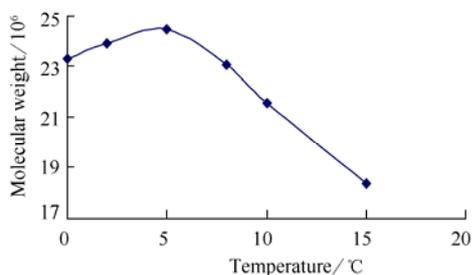


Fig. 1 Effect of initiation temperature on molecular weight

decomposition of the initiator, speed up the polymerization rate and also promote side reactions, thus reducing star-polymer molecular weight and viscosity, which is consistent with the kinetics of usual free radical polymerization reactions. The optimized initiation temperature for polymerization is determined as 5 °C.

2.1.3 Effect of acrylamide mass fraction

During the polymerization between the graft β -CD functional monomer and acrylamide, acrylamide is the monomer with higher polymerization activity. In a large range of acrylamide mass fractions, the reaction can all get the star-polymer with higher molecular weight.

Fig. 2 shows that with the variation of acrylamide monomer mass fraction, the molecular weight of star-polymer would increase first and then decrease. When the mass fraction of acrylamide monomer is lower, with an increase of monomer mass fraction, the polymerization degree of the star-polymer increases and the molecular weight of star-polymer also increases. When the mass fraction of acrylamide monomer is too high, the molecular weight of the star-polymer will decrease, the reason being that during the polymerization step with a high mass fraction of acrylamide monomer, the monomer polymerization heat will be greater and the released heat can not be exported in time. This results in the acceleration of the initiation rate, increasing the chain transfer reaction such that molecular chains between macromolecules rupture. Therefore, the molecular weight of star-polymer decreases. The higher the mass fraction of acrylamide monomer is a significant factor of this effect. When the mass fraction of acrylamide monomer is 25%, star-polymer has the largest molecular weight.

2.1.4 Effect of graft β -CD functional monomer mass fraction

During the polymerization process between the graft β -CD

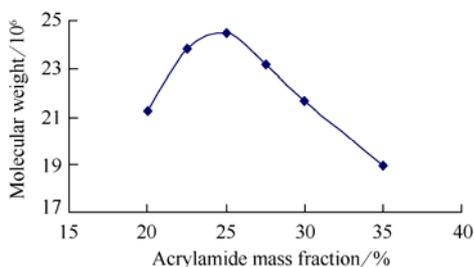


Fig. 2 Effect of acrylamide mass fraction on molecular weight

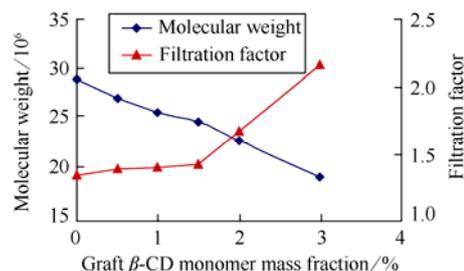


Fig. 3 Effect of graft β -CD functional monomer mass fraction

functional monomer and acrylamide, the polymerization activity of the graft β -CD functional monomer is lower. Figure 3 shows that, with the increases in graft β -CD functional monomer mass fraction, the molecular weight of the star-polymer will decrease gradually, while the filtration factor increases gradually. When the mass fraction of the graft β -CD functional monomer is larger than 1.5%, the filtration factor of star-polymer will be greater than 1.5, which cannot satisfy the requirement of the polymer to be used for oil displacement. Results suggest the optimized polymerization reaction graft β -CD functional monomer mass fraction to be 1.5%.

2.1.5 Effect of Azobisisobutyronitrile mass concentration

Figure 4 shows that, the molecular weight of star-polymer increase first and then decreases with the increasing of azobisisobutyronitrile mass concentration. The reason is that when azobisisobutyronitrile mass concentration is lower, free radicals decrease in the late period of copolymerization between graft β -CD functional monomer and acrylamide a chain termination reaction will dominate, and the reaction will be incomplete, resulting in a decrease in the molecular weight of the star-polymer. With an increase in azobisisobutyronitrile mass concentration, the free radicals in the late period will also increase, molecular chain length will increase further, thus molecular weight increase. When the azobisisobutyronitrile mass concentration is over 50 mg/L, more free radicals will be generated during the late period of polymerization between the graft β -CD functional monomer and acrylamide, the temperature of the reaction system will increase, resulting an increase of chain termination reaction rate, speeding up the polymerization reaction leading to kinetic chain length reduction and decreasing the molecular weight of the star-polymer. Therefore the optimal value for azobisisobutyronitrile concentration during copolymerization is 50 mg/L.

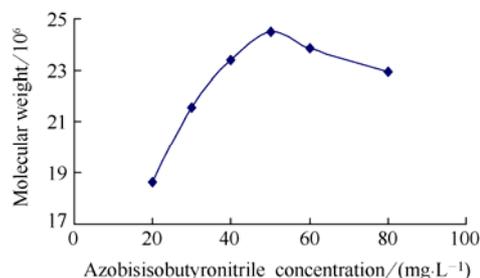


Fig. 4 Effect of Azobisisobutyronitrile mass concentration on molecular weight

2.1.6 Effect of hydrolysis temperature

If hydrolysis occurs at low temperatures, because star-polymers have a larger molecular weight, it will have a larger gel elasticity and hardness on a macroscopic scale, which is not favorable for hydrolysis agents to disperse into the gel or disperse slowly. The products are generally the mixture of which hydrolysis occurs on the surface but not in the internal. Because non-water dissolution has a much lower contribution to molecular weight than water dissolution, therefore the polymer presents as a lower molecular weight product. However, this situation could be offset by prolonging hydrolysis time. When the temperature is lower than 100 °C, the macromolecules of star-polymer gel are in a frozen state, but the temperature increase did not cause any mutual movement between macromolecules, only molecular vibration occurs. When temperature is over 100 °C, significant macromolecule decomposition occurs.

The continuous volatility of water molecules under high temperature let the amides in large molecular chains get closer and closer, eventually leading to crosslinking amidization, resulting in the increase of insoluble content and leading to a decrease in molecular weight.

In addition, if considering the reaction from an energy conservation point of view, the hydrolysis temperature should be as low as possible. Fig. 5 shows that with the increasing temperature, the molecular weight of star-polymer has a trend of increasing first and then decreasing and the molecular weight is the highest when hydrolysis is 90 °C.

2.1.7 Effect of hydrolysis time

If hydrolysis time is too long, the molecular chain can be degraded and the molecular weight reduced. If hydrolysis time is too short, the hydrolysis would not be completed and

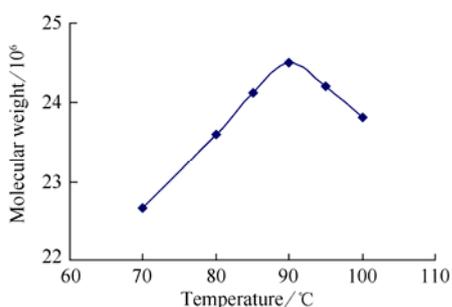


Fig. 5 Effect of hydrolysis temperature on molecular weight

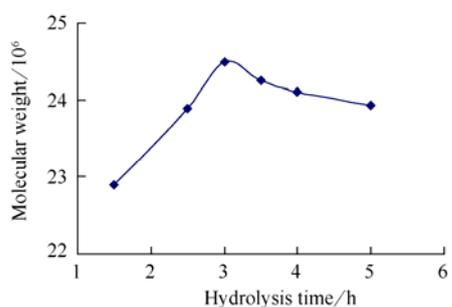


Fig. 6 Effect of water solution time on molecular weight

the molecular weight of the star-polymers would be reduced. It can be seen from Figure 6 that a hydrolysis time of 3 h is optimal.

2.2 Characterization of star-polymer

2.2.1 IR spectra characterization of star-polymer

Figure 7 shows the infrared spectra of star-polymer S07313 samples. In addition to containing a carbonyl characteristic peak (1651 cm^{-1}) and an amide characteristic peak ($-\text{CONH}_2$: 3432 cm^{-1} ; $-\text{C}-\text{N}-$: 1453 cm^{-1} ; $-\text{C}-\text{H}-$: 1324 cm^{-1}), it also contains the graft β -CD functional monomer characteristic peak of $-\text{CH}_2-$ (2856 cm^{-1}) and pyranose ring characteristic peak (1264 cm^{-1}), which prove the presence of the star-polymer S07313 star-nuclear graft β -CD functional monomers.

2.2.2 Topography of star-polymers observed by atomic force microscopy

An Atomic Force Microscope CSPM4000 was used to observe the topography of star-polymer S07313 samples (Fig. 8). The image pixels are 512 \times 512 and image size is 20000.00 \times 20000.00 nm. The surface morphology of star-polymer S07313 is of a star dendritic molecular aggregate, where the main body length is 7500.00–12500.00 nm and the length of the main branches are 200.00–500.00 nm.

2.3 Property evaluation of star-polymer

Daqing oilfield simulated saline was used (total salinity 2350 mg/L, in which the $\text{Ca}^{2+} + \text{Mg}^{2+}$ content is 19 mg/L). Some solution properties, such as physical-chemical informa-

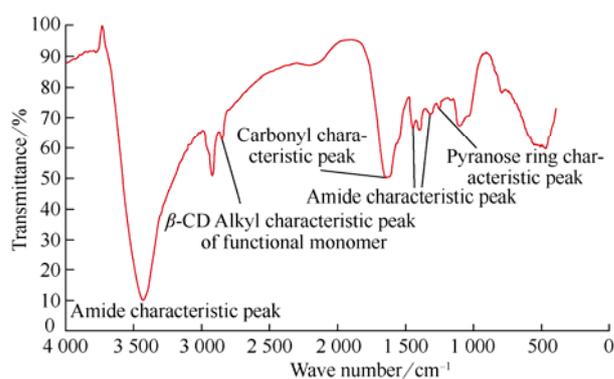


Fig. 7 Infrared spectra of S07313

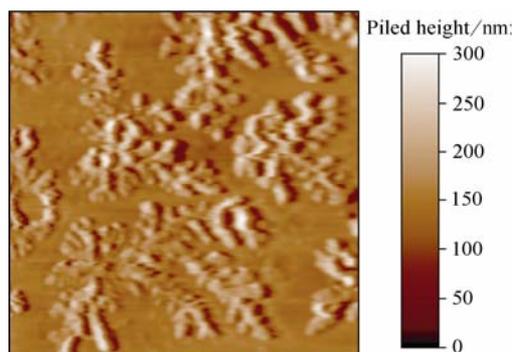


Fig. 8 Topography of star-polymer S07313

Table 2 Physical-chemical property indices of different polymers

Item	Solid content/%	Relative molecular mass/ 10^6	Limiting viscosity number/(dL g^{-1})	Hydrolysis degree/%	Apparent viscosity/($\text{mPa}\cdot\text{s}$)	Filtration factor	Water-insoluble /%	Solution time/h
S07313	88.15	24.5	28.12	24.9	63.9	1.43	0.18	<2.0
Polymer HPAM3000	89.15	30.5	32.49	25.7	58.3	1.49	0.19	<2.0
MO4000	89.98	20.7	25.16	26.8	38.3	1.46	0.15	<2.0
Technology requirement	≥ 88			23–27	≥ 40.0	≤ 1.5	≤ 0.2	≤ 2.0

tion, salt-resistance, alkali-resistance and the compatibility with surfactants were evaluated for star-polymer S07313 and the comparative samples of high molecular weight polyacrylamide HPAM3000 and MO4000. The basic test conditions were: polymer concentration of 1000 mg/L, salinity 2350 mg/L, 45 °C, BROOKFIELD viscometer UL rotor, shear rate 7.34 s^{-1} .

2.3.1 Physical-chemical properties of star-polymer

It can be seen from Table 2 that the molecular weight of star-polymer S07313 is 2.450×10^4 , its solid content, hydrolysis degree, filtration factor, water insoluble, dissolution rate and apparent viscosity all meet the technical requirements for oilfield application.

2.3.2 Salt- and alkali (NaOH)-resistance of star-polymer

It can be seen from Figure 9 that in 6 kinds of brine with increasing salinity (2350 mg/L, 7350 mg/L, 12350 mg/L, 32350 mg/L, 52350 mg/L, 102350 mg/L), the apparent viscosity of all 3 samples decreased with the increase in salinity. When salinity is 102350 mg/L, the apparent viscosities of salt- and alkali-resistant star-polymers S07313, HPAM3000 and MO4000 are respectively 29.70 mPa·s, 18.50 mPa·s and 12.10 mPa·s.

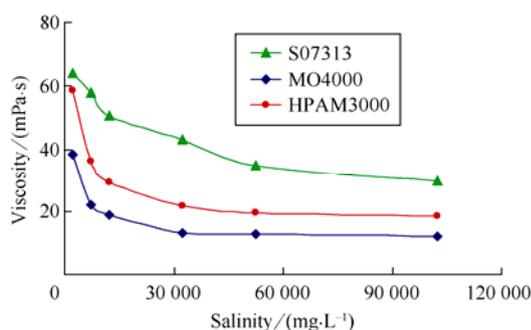
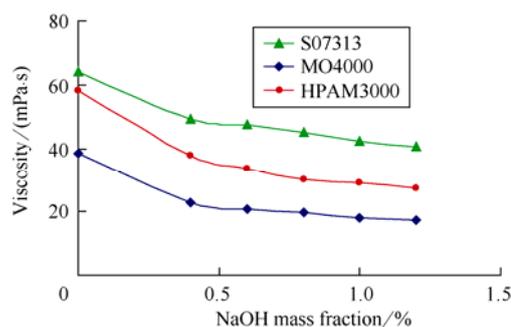
**Fig. 9 Relationship between polymer viscosity and salinity**

Figure 10 shows that when an alkaline solution with NaOH mass fraction of 0, 0.4%, 0.6%, 0.8%, 1.0% and 1.2% respectively are added into the Daqing simulated saline, the apparent viscosities of all three samples will decrease with the increase in NaOH mass fraction. When the NaOH mass fraction is 1.0%, the apparent viscosities of salt- and alkali-resistant star-polymers S07313, HPAM3000 and MO4000 are respectively 42.40 mPa·s, 29.20 mPa·s and 18.00 mPa·s. Because the star-shaping of the main chain for star-polymer S07313 can effectively increase the rigidity of the polymer molecular chain, the curling of the polymer molecular chain became difficult, thus the hydraulic radius of molecular chain rotation increases, so the viscosity increment ability and salt- and alkali-resistant ability were improved significantly.

2.3.3 Compatibility between star-polymer and surfactant

The experiment data comparing S07313, HPAM3000, MO4000 and surfactant alkylbenzene sulfonate are shown in Table 3. A crude oil from Daqing (the Fourth Plant) was mixed with alkylbenzene sulfonate and S07313, HPAM3000 and MO4000 solutions and the resultant measured Interfacial tension are $2.51 \times 10^{-3} \text{ mNm}^{-1}$, $7.23 \times 10^{-3} \text{ mNm}^{-1}$ and $7.62 \times 10^{-3} \text{ mNm}^{-1}$ respectively. At a alkylbenzene sulfonate concentration of 2000 mg/L, temperature of 45 °C, and adding

**Fig. 10 Relationship between polymer viscosity and NaOH content****Table 3 Compatibility between star-polymer and surfactant**

Polymer sample	Polymer concentration/(mg L^{-1})	Alkylbenzene Sulfonate concentration/(mg L^{-1})	System viscosity/($\text{mPa}\cdot\text{s}$)	Interfacial tension / ($10^{-3} \text{ mN m}^{-1}$)
S07313	1000	2 000	35.80	2.51
HPAM3000	1000	2 000	29.80	7.23
MO4000	1000	2 000	18.90	7.62

Note: Viscosities were determined using BROOKFIELD viscometer, UL rotor, at the condition of 6 r / min.

alkaline with 1.0% NaOH mass fraction into the Daqing simulated saline, the interfacial tension between Daqing crude oil and alkylbenzene sulfonate was measured as 7.49×10^{-3} mNm⁻¹. This shows that the compatibility between the star-polymer S07313 sample and alkylbenzene sulfonate surfactant is obviously better than that of super-high molecular mass polyacrylamide HPAM3000 and MO4000.

3 Conclusions

A salt- and alkali-resistant star-polymer was made by graft β -CD functional monomer and acrylamide monomer copolymerization and post-hydrolysis. The optimum parameters: azobisisobutyronitrile concentration of 50 mg/L, the mass fraction of acrylamide 25%, the graft β -CD functional monomer mass fraction of 1.5%, initiating temperature 5 °C, hydrolysis temperature 90 °C and hydrolysis time of 3 hours.

AFM morphology and infrared spectroscopy results showed that salt- and alkali-resistant star-polymer S07313 contains star-nuclear graft β -CD functional monomer, which proves it is a polymer with a star structure.

The evaluation of physicochemical properties of salt- and alkali-resistant star polymers showed that the molecular weight for polymer S07313 is 2450×10^4 , the polymer's solid content, hydrolysis degree, filtration factor, water-insoluble matter, dissolution rate and apparent viscosity and other parameters can all meet the technology requirement for an oil-field application index.

The solution properties of salt- and alkali-resistant star-polymer S07313, such as salt resistance, alkaline resistance and the compatibility with surfactants are obviously better than that of super-high molecular weight polyacrylamide HPAM3000 and MO4000.

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