

文章编号:1000-4092(2022)01-076-06

超低渗透砂岩 SiO_2 纳米颗粒吸附滞留特征*

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摘要: 纳米颗粒因其独特的纳米效应在提高原油采收率具有广泛的应用前景, 但超低渗储层孔喉细小, 纳米颗粒的吸附滞留对其储层物性影响较大。基于 SiO_2 纳米流体在超低渗岩心中的驱替实验, 结合紫外可见分光光度实验测试纳米颗粒在岩心中吸附量, 并采用扫描电镜观察了驱替结束后岩心切片。研究结果表明, 随着纳米流体质量分数(0.01%~0.50%)的增加, 岩心注入压力升高, 纳米颗粒滞留率增大(7.60%~87.50%)、渗透率损失率最高可达96.46%。后续NaCl溶液驱替仅可带走少许吸附不稳定的游离态纳米颗粒, 但未明显缓解吸附滞留情况, 纳米颗粒已在岩心中形成了有效封堵。为了不影响后续流体的注入, 超低渗砂岩注入 SiO_2 纳米流体的质量分数不能大于0.01%。驱替结束后岩心切片的SEM扫描图像显示, 纳米颗粒集中吸附在岩心前段的孔喉和基质表面, 占据流体渗流通道, 引起孔喉结构变化。纳米流体浓度越大, 颗粒聚集现象越明显。

关键词: 纳米颗粒; 超低渗; 吸附; 封堵

文献标识码: A DOI: 10.19346/j.cnki.1000-4092.2022.01.014

中图分类号: TE357

开放科学(资源服务)标识码(OSID):



纳米流体具有良好的提高原油采收率的能力, 纳米颗粒作为添加剂可减少聚合物和表面活性剂的吸附; 作为驱替剂时, 可实现岩石表面润湿反转、增强泡沫和乳状液稳定性、改善水油流度比、减小油水界面张力等^[1-9]。超低渗储层油气储量丰富但物性较差, 开采难度大, 表现为发育微-纳米级孔喉、孔隙连通性差、流体渗流阻力大等^[10-14]。纳米流体独特的纳米效应使之能够进入超低渗储层中, 由于其较强的吸附性, 高浓度时势必引起超低渗储层结构变化, 导致孔隙空间减小、渗透性降低, 进而对后续流体的注入造成影响^[15-18]。Sun等^[19]研究了添加SDS和 SiO_2 纳米颗粒的泡沫在不同渗透率填砂模型中的动态表面性质和纳米颗粒的滞留特征, 认为吸附过程中SDS和纳米颗粒存在竞争关系, 纳米颗

粒降低了混合系统的吸附速率, 纳米颗粒的滞留量随模型渗透率增大而减少。Liu等^[20]研究发现在碱性和低盐度条件下有利于 SiO_2 纳米颗粒在碳酸盐岩储层中运移, 纳米颗粒在基质中的吸附是多层的。Yuan等^[21]通过考虑最大吸附浓度、脱附浓度等必要参数建立了不同浓度纳米颗粒在油湿砂岩中流动引起渗透率变化的模型, 可用于实验分析纳米颗粒吸附、应变、脱附行为对地层造成的损伤。本文通过 SiO_2 纳米流体在超低渗岩心中的驱替实验, 结合紫外可见分光光度计提出了测试纳米颗粒在岩心中吸附量的方法, 测试不同注入阶段的压力、滞留率、渗透率等参数, 定量表征纳米颗粒在岩心中的吸附滞留特征, 并通过SEM扫描电镜技术观测 SiO_2 纳米颗粒在超低渗砂岩内的吸附形态, 解释吸

* 收稿日期: 2021-04-19; 修回日期: 2021-05-26。

基金项目: 国家科技重大专项“低渗-特低渗油藏纳米流体对储层油水相渗特性影响及其驱油效果研究”(项目编号2017ZX05009004-002)。

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附-脱附机理,从而为纳米流体提高采收率技术的应用提供一定的理论指导。

1 实验部分

1.1 材料与仪器

SiO₂ 纳米颗粒为亲水型,比表面积(BET)200 m²/g、悬浮液 pH 值(4%)9.7、密度 1.15 g/cm³,粒子带负电,平均粒径 10 nm,阿法埃莎(Alfa Aesar[®])化学有限公司;天然超低渗砂岩岩心,φ 2.5×10 cm,胜利油田某区块;氯化钠,纯度 99.5%,上海阿拉丁(Aladdin[®])生化科技股份有限公司;实验用水为超纯水。

S-4800 型扫描电子显微镜(SEM),日本日立公司;Thermo Scientific GENESYS[™] 10S 型紫外可见分光光度计,赛默飞世尔科技(中国)有限公司;岩心驱替实验装置系统,包括岩心夹持器、ISCO 泵等。

1.2 实验方法

1.2.1 SiO₂ 纳米流体注入实验

(1)取用 4 块来自胜利油田某区块的天然超低渗砂岩岩心,清洁干燥后以 0.5 mL/min 的速率进行水驱 3 h 测试岩心渗透率 K_1 。岩心基本参数如表 1 所示。

表 1 天然超低渗砂岩岩心的基本参数

岩心 编号	纳米流体 质量 分数/%	长度/ cm	直径/ cm	水驱渗透率 $K_1/(10^{-3} \mu\text{m}^2)$	孔隙度/ %
①	0.01	9.072	2.47	0.38	19.58
②	0.05	9.090	2.46	0.25	19.56
③	0.10	9.472	2.48	0.62	18.59
④	0.50	9.954	2.47	1.98	20.09

(2)向质量分数 3% 的 NaCl 溶液中加入一定量的 SiO₂ 纳米颗粒,并进行超声处理避免颗粒团聚,配制成 4 种不同浓度 SiO₂ 纳米流体;(3)以 0.5 mL/min 的速率分别向 4 块岩心注入 5 PV 的相应浓度的纳米流体并连续收集产出液。每次取 3 mL,利用紫外分光光度计测量产液中纳米颗粒浓度。

(3)以 0.5 mL/min 的速率注入 16 PV 的质量分数 3% 的 NaCl 溶液,记录整个驱替过程岩心两端压差。

利用达西公式计算不同驱替方式下的岩心渗透率 K_2 、 K_3 。由注入前后岩心的渗透率之差与注入

前岩心渗透率之比即 $(K_1-K_{2/3})/K_1$ 计算岩心渗透率损失率。

1.2.2 扫描电镜分析

将 NaCl 溶液驱替结束后的 4 块岩心分别在前、中、后 3 段切片,喷金处理后,利用扫描电镜观察 SiO₂ 纳米颗粒在天然超低渗砂岩岩心中的吸附情况。

2 结果与讨论

2.1 纳米颗粒在超低渗岩心中的吸附与滞留特征

依据朗伯比尔定律, SiO₂ 纳米颗粒对某一波长的光吸收强度与浓度存在定量关系。测量不同质量分数(0.1%~2%)的纳米流体的透光率,绘制浓度-透光率的标准曲线,如图 1 所示。利用紫外可见分光光度计法^[22]测试产出液中 SiO₂ 纳米颗粒的吸光强度,在标准曲线上确定其浓度。

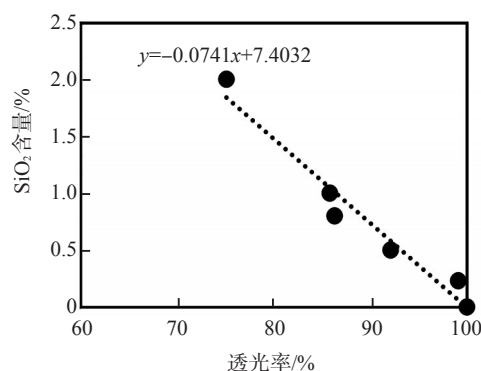
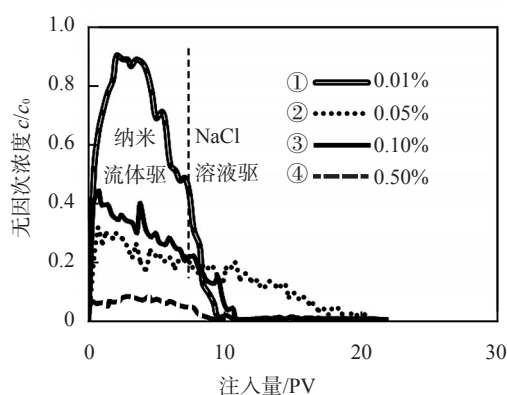


图 1 SiO₂ 纳米颗粒浓度与透光率标准曲线

将超声分散后的纳米流体静置 1 d 后,不同浓度的纳米流体均未出现明显的分层现象,因此在实验过程中不会出现 SiO₂ 纳米流体未进入岩心而颗粒提前大量聚集沉降现象。使用动态光散射粒度分析仪(DLS)测量静置后的纳米流体中纳米颗粒的平均尺寸,质量分数分别为 0.01%、0.05%、0.10%、0.50% 的纳米流体对应的纳米颗粒平均尺寸为 117、135、145、156 nm,由此可见纳米颗粒在 3% NaCl 溶液中分散性较好。

设注入端 SiO₂ 纳米流体的浓度为 c_0 ,产出端为 c ,引入无因次浓度 c/c_0 用以表征纳米颗粒在岩心中的吸附量。图 2 为无因次浓度随注入量变化关系曲线,每条曲线与横轴所围图形面积表示该浓度下未吸附于孔喉中而随流体一起产出的纳米颗粒量。



曲线参数为纳米流体质量分数。

图2 无因次浓度随注入量变化关系曲线

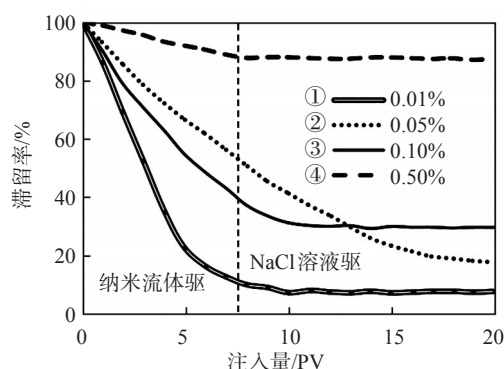
从图2可看出纳米流体浓度越低,图形面积越大,在岩心中吸附滞留率越小。

开始注入纳米流体时,由于纳米颗粒在岩心中吸附不稳定,吸附速率小于解吸附速率,颗粒大多随流体产出,曲线呈上升趋势。随着注入量增加,颗粒与岩石碰撞并发生反应的概率增大,颗粒之间聚集面积增大,并牢牢吸附在基质表面,吸附稳定性增强,流体对已吸附颗粒的冲刷剪应力减小,产出端 SiO_2 纳米颗粒数量逐渐减少。后续NaCl溶液驱替过程中,岩心中仅未完全吸附的自由态纳米颗粒被驱替液带出。

纳米颗粒进入岩石孔喉后,通过静电作用吸附在岩石表面的上;此外由于纳米颗粒含有大量的亲水基团,在岩石表面产生强烈的氢键作用。纳米颗粒在岩石表面发生吸附主要是由于静电和氢键作用^[23]。吸附速率/解吸附速率指单位时间内单位吸附面积上颗粒增加/减少的数量。影响二者的因素很多,如岩石表面粗糙度、纳米颗粒与岩石颗粒间作用力大小、岩石颗粒化学组成、纳米流体及NaCl浓度、多孔介质比表面积等。因为纳米颗粒吸附会导致岩石表面 ζ 电势升高^[24],可通过绘制单位面积上 ζ 电势随时间变化关系曲线衡量二者相对大小。具体数值需通过分子动力学模拟实验结合Langmuir吸附模型计算得出。本文仅以出口无量纲颗粒浓度变化率的正负从宏观上表征吸附速率和解吸附速率的相对大小。斜率为负时,吸附速率>解吸附速率;斜率为正时,吸附速率<解吸附速率。

图3为岩心滞留率随注入量变化曲线。曲线倾斜绝对值随注入量增加逐渐放缓,说明 SiO_2 纳米颗粒

粒滞留率在前期对于注入量变化敏感度大于后期。原因在于基质表面吸附点位不断被占据,岩石和纳米颗粒接触碰撞面积持续减小,发生相互作用而产生吸附的概率降低。整体上, SiO_2 纳米颗粒浓度越大,其在岩心中的最终滞留率越大。②③号岩心对应曲线在纳米流体驱替阶段存在异常是因为水驱渗透率差异较大,故纳米流体浓度和岩心渗透率均是纳米颗粒滞留率的影响因素。



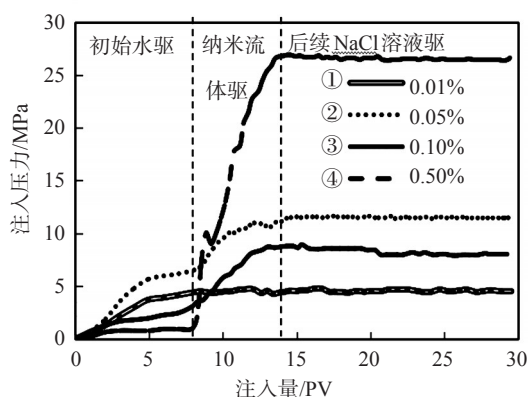
曲线参数为纳米流体质量分数。

图3 SiO_2 纳米颗粒滞留率随注入量的变化曲线

纳米流体驱、后续NaCl溶液驱后岩心的渗透率见表2。由于各天然岩心水驱渗透率存在差异,为消除该影响引入渗透率损失率,用来表征纳米流体和NaCl溶液注入后岩心渗透率的相对变化程度。驱替阶段注入压力随注入量的变化见图4。由表2可知,随纳米流体质量分数的增加,纳米颗粒滞留率和岩心渗透率损失率均增大。只有纳米流体质量分数为0.01%时,二者小于10%,纳米颗粒对岩心的封堵能力最弱。因此在不影响后续流体注入情况下,超低渗砂岩注入 SiO_2 纳米流体的质量分数不能大于0.01%。

表2 不同驱替方式下的岩心渗透率变化情况

岩心	渗透率 $K_s / (10^{-3} \mu\text{m}^2)$	渗透率 $K_s / (10^{-3} \mu\text{m}^2)$	渗透率损失率/%	纳米颗粒滞留率/%
	纳米流体驱后	后续NaCl溶液驱后		
①	0.35	0.35	7.89	7.60
②	0.15	0.15	40.00	17.65
③	0.19	0.20	67.74	29.66
④	0.07	0.07	96.46	87.50



曲线参数为纳米流体质量分数。
图4 注入压力随注入体积的变化

纳米流体驱替阶段注入压力和注入量呈正相关关系。SiO₂纳米流体质量分数为0.01%时,压力增长幅度很小,岩心渗透率基本不变,这是因为纳米颗粒在低浓度时多处于游离态,吸附能力较差,几乎不产生封堵效应。当纳米流体质量分数增至0.5%时,注入压力急剧上升,表明岩心内部高度堵塞,产生憋压,流体流动需要更大的驱动压差。纳米流体驱替后,岩心渗透率均有所下降,后续NaCl溶液的注入并没有缓解堵塞,注入压力近似为水平直线,岩心渗透率损失不可逆。

2.2 SEM图像分析

驱替前岩心的SEM扫描图像如图5所示。驱替结束后的各岩心切片内部SiO₂纳米颗粒吸附情况如图6—图9所示,从左至右依次代表前、中、后3

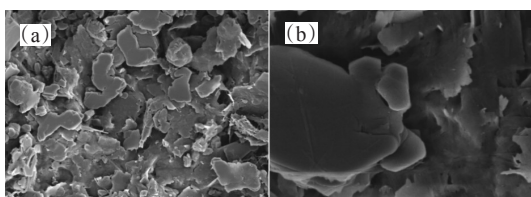


图5 天然砂岩岩心SEM扫描图

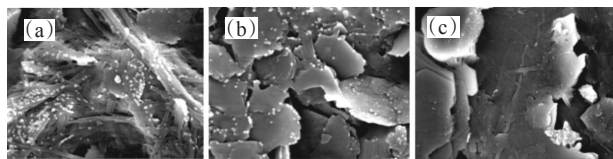


图6 ①号岩心切片纳米颗粒吸附情况(0.01%)

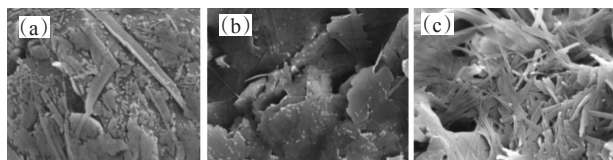


图7 ②号岩心切片纳米颗粒吸附情况(0.05%)

段的切片。

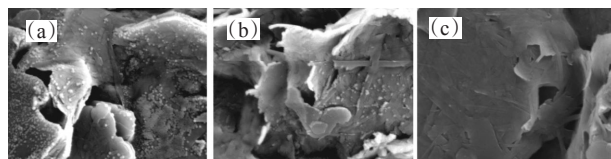


图8 ③号岩心切片纳米颗粒吸附情况(0.10%)

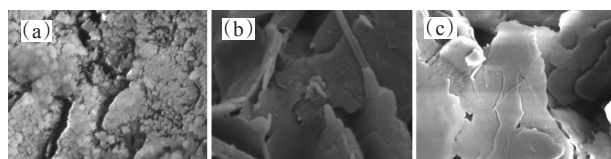


图9 ④号岩心切片纳米颗粒吸附情况(0.50%)

与驱替前岩心相比,纳米颗粒质量分数为0.01%时,驱替后岩心中纳米颗粒总吸附量很少,颗粒分散度高,分布不均匀,未见颗粒大规模聚集现象,纳米颗粒几乎不影响岩心孔喉结构。纳米颗粒质量分数为0.05%时,驱替后岩心中颗粒吸附总量明显增多,分散度降低,开始出现大范围聚集现象,且优先滞留于孔隙空间,其次位于基质表面,孔喉结构发生改变,孔隙空间大幅减小。纳米颗粒质量分数为0.1%时,纳米颗粒在岩心前段的吸附量进一步增大,聚集现象愈发明显,颗粒间排布紧密,导致后续注入难度倍增,中、后段岩心壁面近乎光滑。纳米颗粒质量分数为0.5%时,纳米颗粒已完全覆盖前段岩石基质表面,无法观测到裸露基质和单个存在的纳米颗粒,颗粒之间不规则聚集重叠现象严重,形成大而厚的致密吸附层,形如絮状沉淀,几乎完全堵塞孔喉,表面粗糙度较大。中、后段切片几乎无颗粒吸附而保持初始形态,说明纳米颗粒主要吸附在岩心头部,封堵渗流通道,导致后续颗粒无法向深部运移。

纳米颗粒在砂岩岩心的孔隙和喉道内与其发生相互作用或自身间作用,产生颗粒与孔隙或颗粒与颗粒之间的吸附及黏连,形成吸附层,吸附力大于流体剪切力,致使颗粒在岩心内部滞留,占据流体的有效流动空间。

3 结论

随着纳米流体质量分数(0.01%~0.50%)的升高,纳米颗粒与岩石碰撞并吸附的概率增大,颗粒与基质或颗粒之间相互作用愈发显著,导致注入压

力升高,纳米颗粒滞留率(7.60%~87.50%)增大,后续NaCl溶液驱替不能解除堵塞。

当SiO₂纳米颗粒进入岩心后优先占据孔喉等渗流通道,减小孔喉有效尺寸,通过聚集成块并吸附于基质表面从而减小有效渗透率。因此在不影响后续流体注入情况下,超低渗砂岩注入SiO₂纳米流体质量分数不能大于0.01%。

颗粒主要吸附在岩心前段的孔隙空间,并在基质表面形成不同分散度的吸附层,引起孔喉结构变化。中、后段由于头部堵塞导致颗粒流动性急剧降低而不能形成明显吸附面。

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Adsorption Characteristics of SiO₂ Nanoparticles in Ultra-low Permeability Sandstone

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Abstract: Nanoparticles have a good application prospect in enhancing oil recovery due to its unique nano-effects, but the ultra-low permeability reservoirs have small pore throats, and the adsorption of nanoparticles has a greater impact on the physical properties of the reservoir. Based on the displacement experiments of SiO₂ nanofluids in the ultra-low permeability cores, combined with the ultraviolet-visible spectrophotometer, the adsorption capacity of nanoparticles in cores was tested, and the core slices after displacement were observed by scanning electron microscope. The research results showed that when mass fraction of nanofluids increased from 0.01% to 0.50%, the core injection pressure increased, and the retention rate of nanoparticles increased, being of

7.60%—87.50%, as a result, the permeability loss rate could reach up to 96.46%. The subsequent displacement of NaCl solution only took away a few unstably adsorbed free nanoparticles, but did not alleviate the adsorption obviously, because of the formation of effective plugging in the rock core. In order not to affect the subsequent injection of fluids, the mass fraction of SiO₂ nanofluids injected into ultra-low permeability sandstone should not exceed 0.01%. The scanning images of core sections after displacement demonstrated that the nanoparticles were concentrated on the pore throat and matrix surface at the front of the core, occupying the fluid seepage channel, which caused structural changes in the pore throat. As the concentration of nanofluids increased, the particle aggregation phenomenon became more obvious.

Keywords: nanoparticle; ultra-low permeability; adsorption; plugging

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Evaluation and Application of Polymer Microsphere and Surfactant Compound Profile Control and Flooding System

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Abstract: According to the characteristics of Changqing low permeability reservoir, polymer microsphere and surfactant compound profile control and oil recovery technology was proposed. Polymer microsphere was prepared by acrylic acid, 2-acrylamide-2-methylpropanesulfonic acid, 2-mercaptobenzoic acid, ammonium persulfate and sodium bisulfite. Surfactant complex system was prepared by alkanolamide polyoxyethylene polyether sulfonate and coconut oil fatty acid diethanolamide. The oil-water interfacial tension of surfactant and surfactant/polymer microsphere mixture was studied, the profile control and displacement performance of polymer microsphere and mixture was investigated, and the injection mode of compound flooding was optimized. Finally, it was applied on Ansai oilfield. The results showed that the initial particle size of polymer microsphere was generally 50—300 nm. It had hydration expansion characteristic with expansion ratio of 20—100 times. The aggregation characteristic of microspheres during hydration and expansion was observed by SEM. The microspheres had good dispersion and sphericity, and the particle size distribution was Gaussian normal distribution. The most economical concentration of surfactant was 3 g/L. The viscosity of the mixture increased after adding surfactant to polymer microsphere, and the dispersed phase particle of microsphere shielded the interfacial activity and micelle formation ability of surfactant, which resulted in the decrease of interfacial tension and was not conducive to surfactant flooding. Polymer microsphere solution had good plugging performance to core. When the mass concentration of microsphere was greater than 4 g/L, the plugging rate was about 80%. The oil displacement efficiency of polymer microsphere and surfactant by slug injection mode with volume ratio of 1:1 was better than that by mixed injection of two systems. The application effect of this technology in Ansai oilfield was good, and the cumulative oil increase was 3576 t.

Keywords: nano polymer microsphere; surfactant; compound profile control and flooding; enhanced oil recovery; Ansai oilfield

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rapid rise of water cut, so water control is urgently needed. Based on the reservoir features and wells production performances, the terpolymer was prepared using acrylamide, 2-acrylamide-2-methylpropanesulfonic acid and dimethyldiallyl ammonium chloride for selective water shutoff in horizontal well. The effects of crosslinking agent *N*, *N*-methylene bisacrylamide on the strength and swelling rate of water shutoff agent were studied. The injection performance and plugging ability of water shutoff agent were investigated. On the basis of laboratory experiment and well performance, the oilfield trials of water shutoff for 6 horizontal wells were carried out in Kunbei oilfield. The experiment results showed that the amount of crosslinking agent had great influence on the strength and swelling rate of water shutoff agent. When the dosage of crosslinking agent was 1.5% of the total mass of monomer, the strength of water shutoff agent was moderate. It could be stretched, and the swelling rate reached the highest. The average size of water shutoff agent particle was 420 μm, which could form effective plugging in the fracture of Kunbei reservoir. After injecting water shutoff agent into sandpack column, the injection pressure of subsequent water flooding increased by 5.06 times, and the plugging rate of water flooding was 83.5% and that of oil flooding was 20%, showing a good selectivity to oil and water. The water shutoff agent was applied in Kunbei oilfield and the remarkable effect of increasing oil and reducing water was gained.

Keywords: glutenite oil reservoir; horizontal well; terpolymer; selective water shutoff agent; Kunbei oilfield