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印尼油砂性质分析及水基分离研究

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摘要: 对印尼油砂的基本性质如油砂的组分、砂粒的粒径分布、砂粒的润湿性及沥青的四组分进行分析,测试了沥青、黏土在不同 pH 值下的 Zeta 电位,考察了二价金属离子 Ca^{2+} 对沥青、黏土 Zeta 电位的影响,同时对印尼油砂进行水基提取分离。分析结果表明,印尼油砂含油率高达 24.9%,砂粒粒径较小,砂粒表面为油润性,沥青中重质组分含量较高。Zeta 电位测试表明,Ca^{2+} 的存在使沥青与黏土表面电位均变正,不利于油砂的分离。水基提取结果显示,印尼油砂的提取率为 46.0%,泡沫质量较差。为了提高沥青收率,应探索适宜的表面活性剂对油砂进行预处理从而改变砂粒的润湿性或直接采用溶剂法开采印尼油砂。

关键词: 印尼油砂; 水基提取; 润湿性; 沥青四组分; Zeta 电位
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随着全球范围石油需求的不断增长及石油价格的不断攀升,对非常规油气资源进行开发和利用具有重大的战略意义[1]。油砂又称沥青砂,是由沥青、砂粒及富矿黏土和水组成的混合物[2],是一种重要的非常规油气资源。根据美国地质调查局(USGS)的研究,世界范围内油砂可采资源量约 6.51×10^{11} 桶,约占世界石油资源可采总量的 32%。全球油砂资源分布很不均衡,主要分布在北美洲、前苏联国家、拉丁美洲及加勒比海地区。此外,印度尼西亚油砂资源丰富,位于其东南的布敦岛,沥青资源储量高达 3.0×10^{9} t,具有很大的开发价值[3-4]。

水基提取技术(WBEPs)是一种重要的油砂分离技术,是目前露天开采油砂的主要方式之一[5]。其特点是沥青收率高、污染小、投资少,因此是油砂开采的首选技术。水基提取技术的成功应用十分依赖油砂自身的性质,特别是油砂中砂粒表面的润湿性。当油砂中砂粒等固体颗粒表面为水润性时,沥青容易从砂粒表面脱附,沥青收率较高;当油砂中砂粒等固体颗粒表面为油润性时,沥青难以从砂粒表面脱附,沥青收率较低、泡沫质量较差[6]。由于油砂的开采、分离方法与其自身性质具有密切的关联[7],因此开展油砂性质分析对于探索其分离技术具有重要的指导意义。本文主要对印尼油砂的组成、砂粒粒径分布、砂粒的润湿性、沥青四组分进行分析,并对沥青、黏土的 Zeta 电位进行测试,考察二价金属离子 Ca^{2+} 的影响,最后采用水基提取技术处理印尼油砂。通过研究油砂性质与其水基提取效率的内在联系,探索适宜的分离技术。

1 实验部分

1.1 实验原料

实验中所用油砂取自印度尼西亚布敦岛,该类油砂黏性极强,是一种质软且呈黑色的大块黏连状物,不易粉碎,为获得小颗粒宜采用冷冻粉碎法处

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理[4,8]。采用加拿大 Alberta 省油砂管理局推荐的标准方法 Dean-Stark 甲苯抽提法对油砂的组成及含量进行分析,结果如表 1 所示。将抽提的沥青置于通风处,待甲苯挥发后用于 Zeta 电位测试。油砂中固体组分粒径分布结果如表2 所示。实验用 KCl(质量分数 >99.999 0%)和 $CaCl_2$(质量分数 >99.996 5%)购自 Alfa Aesar 公司。NaOH(分析纯)和 $NaHCO_3$(分析纯)购自西陇化工股份有限公司。实验中所用水为超纯水(18.25 MΩ·cm)。

表 1　印尼油砂的组成分析

Tab. 1　Composition of Indonesia oil sands

油砂组分	沥青	水	固体组分
质量分数/%	24.9	3.2	71.9

表 2　印尼油砂砂粒的粒度分布

Tab. 2　Grain-size distribution of Indonesia oil sands

粒径范围/μm	<50	50 ~ 106	106 ~ 250	>250
质量分数/%	52.4	14.7	24.4	8.5

1.2　沥青四组分分析方法

沥青组成复杂,根据其组成极性和分子量依次增大而分为饱和烃、芳香烃、胶质和沥青质 4 种组分[9]。其中,沥青质是沥青中极性最大的组分,它易溶于甲苯等芳烃溶剂而难溶于庚烷、戊烷等短链链烃[10]。胶质与沥青质结构相似,因此容易发生团聚而难以分离,但胶质溶于短链链烃。为此,在组分分析时通常先分离出沥青中的沥青质。正庚烷沉淀法是定量分析沥青质含量的常用分析方法。采用此方法时,首先准确称取 2.0 g 的沥青于圆底烧瓶中,然后加入 80 mL 的正庚烷于室温下搅拌溶解 2 h,静置 24 h。将上层液转移至离心管中,在 15 000 r/min 转速下离心 30 min 后取出离心管,将上清液吸出,沥青重新转移至烧瓶中,加入正庚烷进行洗涤、离心,重复此操作直至上清液呈无色,从而得到沥青质。对获得的沥青质干燥、称重,计算其在沥青中的质量分数。对收集的上清液进行减压蒸馏、干燥,从而得到不含沥青质的三组分(Maltenes)。采用柱色谱法对该三组分进行定量分析。柱色谱分析时采用 60 cm × 2 cm 的玻璃柱,柱下半层填充 15 g 中性氧化铝,上半层填充 20 g 硅胶。填料在使用前进行预处理,氧化铝在 400 ℃下活化 8 h,对硅胶采用甲苯/二氯甲烷/甲醇(体积比为 1∶1∶3)混合溶液淋洗,干燥后在 160 ℃下活化 48 h,填料预处理后放置 24 h 后再用。分离时,准确称取 200 mg Maltenes 并加入 10 mL 的正庚烷使其溶解,随后加入 1 g 的硅胶进行拌样,待溶剂挥发完后将样品转移至已被正庚烷充分润湿的色谱柱内,此后分别加入 180 mL 的正庚烷、140 mL 的正庚烷/甲苯(体积比为 2∶1)混合溶液以及 80 mL 的甲苯/二氯甲烷/甲醇(体积比为 1∶1∶1)的混合液淋洗玻璃柱,并分别收集得到饱和烃、芳香烃、胶质组分。将收集的冲洗液进行减压蒸馏,去除溶剂,干燥后称量,计算其在 Maltenes 中的含量[11-13]。

1.3　砂粒表面润湿性分析

砂粒等固体颗粒表面的润湿性是影响沥青从固体表面上脱附的重要因素,是决定油砂水基提取效率的重要性质[14]。为此,采用油水法和压片法[15]对印尼油砂砂粒表面的润湿性能进行评价。

1.3.1　**砂粒样品的制备**　采用溶剂洗涤法获取印尼油砂中的砂粒组分。称取印尼油砂 100 g 于圆底烧瓶中,加入适量的甲苯,在机械搅拌下将油砂溶解后静置,将上层液转移至离心管中,在 15 000 r/min 转速下离心 30 min,吸出上清液后,砂样转移至烧瓶中再次洗涤,重复上述操作至离心后上清液呈无色。随后分别加入乙醇、去离子水洗涤至无色,室温下干燥后,筛出粒径小于 50 μm 细微粒组分,标记为 A。取适量印尼油砂微粒组分 A 在 500 ℃下灼烧 4 h 获得完全亲水性微粒组分 B。

1.3.2　**油水法**　各取 A、B 两种微粒组分样品 0.2 g 分别放入样品瓶中,先加入 4 mL 水(或 4 mL 矿物油)使微粒完全浸润,随后加入 4 mL 矿物油(或 4 mL 水),充分震荡 10 min,静置 5 min 后进行拍照。移去油相、水相,干燥后获得水相中的细微粒组分,称重,计算其含量。在实验过程中考察了矿物油与水加入顺序对细微粒组分在油水两相中分配的影响,先加入水相时,2 种细微粒分别标记为 A_1 和 B_1,先加入油相时则分别标记为 A_2 和 B_2。

1.3.3　**压片法**　各取 A、B 两种微粒组分样品 2.0 g 分别放入直径为 25 mm 的模具内,施加 20 MPa 的压力,保持 2 min 后取出压片。随后在接触角测量仪(JC2000D3,上海中晨)上测试水滴在压片表面的渗透时间。测试中通过注射器将体积约为 6.6 μL 的水滴置于压片表面,记录水滴完全渗透于压片中所需要的时间。为减小实验误差,每组样品均测试 3 个压片,每片样品测试 5 次,计算后取平均值。

1.4　Zeta 电位测量

Zeta 电位是胶体颗粒表面的重要性质,Zeta 电位的高低及带电性质对砂粒等固体组分与沥青之间

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的相互作用具有重要影响[16]。为测量沥青表面的Zeta电位,将印尼沥青在超声条件下分散于超纯水中,从而获得沥青分散水溶液。为制备细微粒砂样悬浊液,取少许粒径小于50 μm的印尼油砂细微粒组分于烧杯中,加入超纯水搅拌,静置1 h取上层浑浊液,进一步的超声,得到黏土悬浊液。测试中,溶液中KCl浓度为1 mmol/L(mM),分别测定不同pH值以及不同Ca^{2+}浓度下沥青、黏土悬浊液的Zeta电位。测试温度为(25±1) ℃。

1.5 水基提取法

印尼油砂水基提取过程在浮选槽内进行。加入900 mL超纯水,利用$NaOH/NaHCO_3$调节其pH值至8.0~8.5,然后加入300 g油砂,混合均匀后在60 ℃下浸泡1 h。在1 500 r/min下搅拌5 min,随后通入气泡(120 mL/min)并收集沥青泡沫。将得到的沥青泡沫转移至Dean-Stark装置内,采用甲苯洗涤,分离出沥青、砂和水,并计算得到沥青的收率和泡沫质量。

2 结果与讨论

2.1 沥青四组分分析

采用正庚烷沉淀法定量分析印尼油砂沥青中沥青质的含量,结果显示,其质量分数为18.3%。采用柱色谱进一步分析Maltenes中三组分的含量。其中,饱和烃质量分数为24.6%,芳香烃质量分数为17.6%,而胶质质量分数为52.6%(注:在柱色谱分析中,部分组分由于极性极强,在填料表面产生不可逆吸附形成柱残留[17]。)

与国内油砂沥青各组分含量相比,印尼沥青中饱和烃含量偏低,而重质组分如胶质、沥青质的含量较高[18-19]。胶质、沥青质的含量是影响沥青黏度的重要因素[20],而沥青黏度对油砂水基提取效率具有重要影响。因此,对于高胶质、沥青质含量的印尼油砂,其水基分离难度较大。

2.2 砂粒润湿性

2.2.1 微粒组分在油水两相中的分布 采用油水法评价微粒组分的润湿性时,样品在水相的分配越多,说明样品的亲水性越强。各微粒组分样品在油水两相中的分配情况如图1所示。从图1可以看出,对于先加入水相的微粒组分A_1,油相与水相均表现浑浊;而先加入油相的微粒组分A_2,水相较A_1来说变得相对澄清,油相浑浊度增加,说明细微粒组分更多地分布到油相中。出现这一现象可能是油相的优先加入导致细微粒组分的优先润湿,油相中的微粒组分含量相对增加。而对于高温灼烧过的表面具有强润湿性的微粒样品B,无论先加水相(B_1)还是油相(B_2),微粒组分均大部分沉于水相底部。

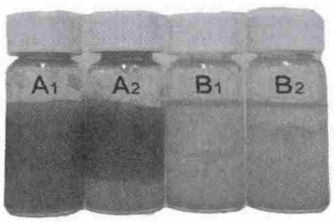

图1 各种微粒砂样在油水两相中的分配
Fig. 1 Distribution of different grain-size sand in water and mineral oil

对A、B两种微粒组分在水相中的分布作定量分析。印尼油砂细微粒组分A,优先加入水相时,微粒组分在水相中的质量分数为81.6%,而当优先加入油相时,微粒组分在水相中的质量分数则为24.4%,说明油相、水相的优先加入对砂粒在水相中的含量影响很大。高温灼烧过的细微粒组分B,优先加入水相与优先加入油相的砂粒在水相中的质量分数分别为98.3%、96.4%,说明经过高温灼烧过的细微粒组分具有强的亲水性,而油相、水相的加入顺序对其影响基本可以忽略。

2.2.2 压片法测量微粒表面的润湿性 压片法则是利用水滴在重力作用下在压片表面的渗透时间来考察微粒的润湿性,渗透时间越长,说明水滴对样品的亲润性越差,样品的疏水性越强。实验测得水滴在印尼油砂细微粒组分A压片表面和经过高温灼烧过的印尼油砂细微粒组分B压片表面的渗透时间分别为105 s和3 s。从这一结果可以看出,印尼油砂中细微粒组分表现出较强的油润性,而经过高温灼烧后,则表现出完全亲水性。

根据相关研究[15],油相、水相的优先加入顺序对弱亲水性或弱亲油性细微粒分散的影响较大。从油水法的实验结果初步判断印尼油砂的细微粒组分为弱亲油性,同时结合压片法中2种油砂细微粒组分表面的渗透时间进一步判断印尼油砂为油润型油砂。

2.3 Zeta电位

图2是沥青表面Zeta电位随溶液pH值和Ca^{2+}浓度的变化曲线。从图2可以看出,随着pH值的增加,沥青乳状液的Zeta电位不断减小,变得更负。溶液pH值在3到6区间沥青乳状液的Zeta电位变化最大,由-47.8 mV降低到-83.0 mV。而当沥

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青乳状液中 Ca^{2+} 含量增加时,沥青表面电负性变小,即 Zeta 电位增大。引起这一现象的原因是 Ca^{2+} 的加入会压缩沥青表面双电层,Ca^{2+} 从扩散层进入紧密层,吸附在沥青表面,中和沥青表面的阴离子、降低表面电荷密度,导致沥青的 Zeta 电位绝对值变小[21]。

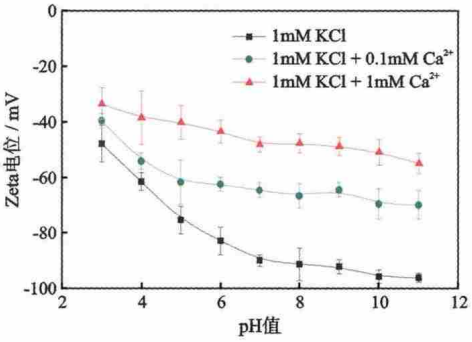

图 2　沥青 Zeta 电位随溶液 pH 值及 Ca^{2+} 浓度变化曲线
Fig. 2　Effects of pH and calcium ion concentration on Zeta potential of bitumen droplet

图 3 是黏土颗粒表面 Zeta 电位随溶液 pH 值和 Ca^{2+} 浓度的变化曲线。从图 3 可以看出,随着溶液 pH 值的升高,黏土表面的 Zeta 电位不断降低,变得更负,pH = 3 及 pH = 11 时的 Zeta 电位分别为 −19.7 mV、−37.67 mV。由于 pH 值的升高,黏土片端部的 Al − OH 和 Si − O 键因电离或断键使得 OH^-、O^{2-} 离去,黏土颗粒端部带的正电荷较黏土层面带的负电荷少,黏土表面电荷为负值,导致 Zeta 电位值更负[22]。随着溶液中 Ca^{2+} 浓度的增加,它在黏土表面发生吸附,降低了其表面的电荷密度,因此黏土的 Zeta 电位绝对值变小。

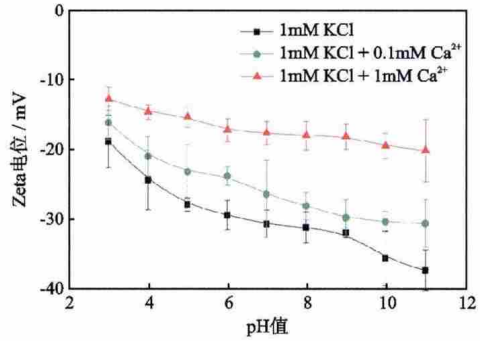

图 3　黏土 Zeta 电位随溶液 pH 值及 Ca^{2+} 浓度变化曲线
Fig. 3　Effect of pH and calcium ion concentration on Zeta potential of clay particle

2.4　印尼油砂水基提取

为了考察印尼油砂砂粒润湿性与油砂水基提取效率之间的内在联系,采用水基提取技术处理印尼油砂,实验结果如图 4 所示。从图 4 可以看出沥青的收率随浮选时间的增加不断上升,在 10 min 时达到 46%,随后趋于稳定,延长浮选时间,沥青的收率没有明显提升。沥青的最终收率低于 50%,说明大部分的沥青难以提取出来。从图 4 还可以看出,泡沫质量在 1.5 min 时为 0.36,5 min 时升至0.48,随后趋于平稳。这一结果说明,随着提取时间的延长,沥青的脱附效率是有所提高的,但是与相关报道相比,泡沫质量较差[6]。在水基提取前,对油砂进行高温浸泡处理,水溶液中的碱性环境会促进沥青表面阴离子表面活性剂的释放,从而降低油水、油固界面张力,同时表面活性剂会影响砂粒表面的润湿性,从而促进沥青的脱附[4-5]。然而,水基提取结果显示在热碱水洗作用下,印尼油砂的沥青收率较低、泡沫质量较差。上述实验结果说明油润性的砂粒表面润湿性能不利于油砂分离,高温碱性环境的浸泡处理未能有效地影响砂粒表面的润湿性能,因此水基提取效率较低。为了有效地解决这一难题,探索能有效改变砂粒润湿性的表面活性剂并用其对油砂进行前处理或采用溶剂提取法提取印尼油砂中的沥青是下一步研究工作的重点和方向。

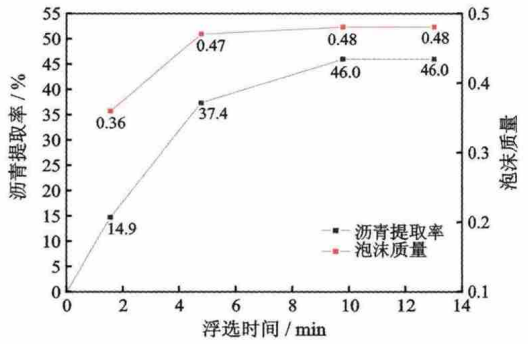

图 4　沥青提取率与泡沫质量随浮选时间的变化曲线
Fig. 4　Effect of flotation time on bitumen extraction rate and bitumen foam quality at 60 ℃ and pH = 8.0 ~ 8.5

3　结　论

(1) 印尼油砂的含油率高达 24.9%,沥青组分分析表明饱和烃含量偏低,重质组分如胶质和沥青质的含量偏高,进而使沥青黏度增大,沥青在砂粒表面的脱附困难,影响沥青收率,同时对沥青的质量也产生一定的影响。

(2) 印尼油砂的砂粒较细,其中细微粒组分(粒径 < 50 μm) 达到 52.4%,而油砂中的细微粒组分在水基提取时容易与沥青发生黏附,不仅使沥青收率降低,还会大大降低沥青的泡沫质量。对其润湿性进行评价后发现,固体颗粒表面表现出弱亲油性。

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(3) 沥青、黏土的 Zeta 电位随着 pH 值的升高变得更负; 当加入不同浓度的 Ca^{2+} 时, Ca^{2+} 在沥青、黏土表面吸附, 使得 Zeta 电位绝对值变小。

(4) 水基提取过程中, 油砂中细微粒组分的含量、砂粒表面的润湿性、沥青的黏度均对沥青的水基提取效率产生一定的影响。采用水基提取法分离印尼油砂时, 沥青提取率仅为 46.0%, 同时泡沫质量较差。为了有效地解决这一难题, 需选择合适的表面活性剂对油砂进行前处理, 改变油砂中砂粒表面的润湿性, 从而提高油砂水基提取效率, 或采用溶剂提取法开采印尼油砂。

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Study on relationship between sedimentary microfacies and water flooding pore volume as well as remaining oil: taking lower V oil layers in Shuanghe Oilfield as an example

Abstract: In order to discuss the relationship between sedimentary microfacies and water flooding pore volume times as well as remaining oil, taking the lower V oil layers in Shuanghe Oilfiled as an example, the mechanism model of water flooding pore volume times is established using numerical simulation method, and the plane quantitative characterization of waterflooding pore volume times and remaining oil distribution are obtained. The inflection point value and the limit value of water flooding pore volume times are determined by analyzing the relationship between water flooding pore volume times and water saturation. The research result shows that, the physical property of the underwater distributary channel sand-bodies developed in the lower V oil layers is the best, that of distributary mouth bar and frontal sheet sand-bodies is secondary, and that of underwater overbank sand-bodies is the worst. Sedimentary microfacies controls the physical property of reservoirs, the difference in the physical property of reservoirs controls the movement of injected water in the plane, the movement of injected water controls water flooding pore volume times, and thereby water flooding pore volume times controls remaining oil distribution, which can be quantitatively illustrated by the combination of the sedimentary microfacies distribution, water flooding pore volume times distribution and remaining oil distribution. The relationship between sedimentary microfacies and water flooding pore volume times as well as remaining oil is important to finding remaining oil enrichment zones and the adjustment of development plan and well pattern to develop the remaining oil in the late period of oilfield development.

Key words: sedimentary microfacies; waterflooding pore volume times; remaining oil; waterflooding development effect; Shuanghe oilfield

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Evaluation of in-use foam drainage agents in Changqing Gasfield and study of a self-generating gas foam drainage agent

Abstract: Four in-use foam drainage agents of Changqing Gasfield are evaluated in laboratory, it is found that they are all of poor condensate resistance. For this reason, a self-generating gas foam drainage agent is developed to extend foaming time, increase lifting force, reduce wellbore fluid density and therefore effectively discharge the water in severe flooded gas wells. The foam drainage agent will generate gas in bottomhole by chemical reaction. The factors influencing the chemical reaction are studied by experiments, and the feasibility of the application of the self-generating gas foam drainage agent in gas fields is discussed. It is shown that, the self-generating gas foam drainage agent is of good suitability to salinity, and compared with in-use foam drainage agents, it is of greater foaming capacity and liquid carrying capacity in Soviet A-2, Sue B-1, Su C-56 wells. Its liquid carrying rate can reach to 53.5%. The basic formulation of the self-generating gas drainage sticks is 41% CQN + 56% CQO + 2.95% CH-1 + 0.05% self-generating gas foam drainage agent.

Key words: foam drainage agent; self-generating gas; condensate resistance; salinity resistance; foam drainage gas recovery

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Properties analysis and water-based separation of Indonesia's oil sands

Abstract: The composition and the grain size distribution of Indonesia's oil sands, the wettability of sand particle and four compo-

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nents of bitumen are analysed, the Zeta potential of asphalt and clay at different pH values is tested, and the effect of Ca^{2+} on the Zeta potential of asphalt and clay are studied. The water-based separation of Indonesia's oil sands is carried out. The analysis results show that, the oil content of the Indonesia's oil sands reaches to 24.9%, its particle size is small, the surface of the oil sands is of oil wettability, and the content of heavy components in the asphalt is high. The presence of Ca^{2+} makes the potential on the surface of asphalt and clay positive, which is not favourable to the separation of the oil sands. The water-based separation of Indonesia's oil sands shows that the extraction rate of the Indonesia's oil sands is 46.0%, and there is poor foam quality. In order to improve the yield of asphalt, the oil sands should be pretreated using suitable surfactants for changing the wettability of the sand particles, or the oil sands is mined directly by using solvent.

Key words: Indonesia's oil sands; water-based extraction; wettability; asphalt four-component; Zeta potential

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Development of low molecular polymer (KGDP) with high-temperature resistance and its application in drilling fluid for deep wells

Abstract: A low molecular mass polymer KGDP with high temperature resistance and salt tolerance is synthesized based on the optimization of molecular structure design and monomer selection, and it is applied to two deep wells with high bottom-hole temperature-Shengke-1 (236 ℃) and Bishen-1 (241 ℃). The thermal stability of its molecule chain, its high temperature resistance, salt tolerance and viscosity reducing effect are evaluated. The results show that, the developed low molecular mass polymer KGDP has good thermal stability, its pyrolytic temperature is up to 330 ℃; its high temperature capacity in water-based drilling fluid slurry can reach to 240 ℃; its viscosity reducing performance is better than similar products at home and abroad; under the bottom-hole temperature of 236 ℃ and 241 ℃, high salinity and high solid content of Shengke-1 and Bishen-1, KGDP plays a role in regulating flow pattern, effectively controls the viscosity and gel strength of water-based drilling fluid to ensure the smooth drilling of two deep wells.

Key words: anti-high temperature low molecular polymer; water-based drilling fluid; viscosity reducer; rheology under high temperature and high pressure; well Shengke-1; well Bishen-1

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Application of octylphenol polyoxyethylene ether carboxylate in viscosity reduction of Tahe heavy oil

Abstract: The viscosity reducer alcohol polyoxyethylene ether carboxylate is synthesized using acetone as solvent. Its salt resistance and thermal stability under different conditions are investigated, and the effects of concentration, oil-water ratio and temperature on the viscosity reducing result of the viscosity reducer and the synergistic effects of the viscosity reducer with cosolvents and polymers are studied. The viscosity-reducing formulation suitable to Tahe heavy oil is obtained. The study results show that alcohol polyoxyethylene ether carboxylate has high thermal stability, and it can maintain excellent interfacial activity in high-salt formation water; increasing the concentration of alcohol polyoxyethylene ether carboxylate, decreasing oil-water ratio and rising emulsification temperature can improve its viscosity-reduction rate to Tahe heavy oil; urea and partially hydrolyzed polyacrylamide could increase the viscosity-reduction effect of alcohol polyoxyethylene ether carboxylate, and to use them as compound cosolvent can reduce the cost of the viscosity reducer. So the compound of alcohol polyoxyethylene ether carboxylate with urea and partially hydrolyzed polyacrylamide is the viscosity reducer formulation suitable to Tahe heavy oil.

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