无机盐离子对石英表面油-水润湿性的影响

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摘要 油藏岩石表面油-水润湿性对原油的开采具有重要影响, 润湿性向亲水转变被认为是低矿化度水驱技术的主要机制, 但是低矿化度水注入后岩石表面发生润湿性转变的微观机理目前依然存在很多争议. 本研究针对极性分子在岩石表面的极性吸附, 研究水中无机盐离子对石英表面润湿性的影响及其作用机理. 研究发现, 相同浓度下, NaCl溶液和KCl溶液的接触角基本相同; 在浓度较低时, CaCl₂溶液使三相接触角降低的幅度比NaCl溶液更大, Na₂SO₄溶液比NaCl溶液效果更明显; 在较高浓度下, Na₂SO₄溶液的接触角与NaCl溶液相似. 当离子浓度大到一定程度时, 不同溶液的接触角都趋于15°左右, 离子使吸附的极性分子基本都脱附, 导致石英表面润湿性向亲水转变. 基于石英粉末的Zeta电位测量和石英片表面的原子力显微镜(atomic force microscopy, AFM)粗糙度分析, 提出了两种机理解释: (1) 癸烷中的十八胺在油水界面质子化带正电, 与水中阴离子相互吸引, 降低了油水界面张力; (2) 水中的阳离子在带负电的石英表面吸附, 使石英片表面负电性减弱, 并置换了部分石英表面吸附的十八胺分子, 使石英表面亲油性减弱.

关键词 润湿性, 接触角, 极性分子, 离子, 多相流动

润湿性作为反映三相界面能相对大小的基本性质, 在众多工业过程中被广泛关注. 在原油开采过程中, 油藏岩石的润湿性会影响毛细管力、微观波及体积和残余油饱和度等^[1~4], 因此合理调控油藏岩石表面的润湿性对提高原油采收率有重要意义. 作为一种功能性水驱技术, 低矿化度水驱技术通过对注入水中的离子种类和浓度配比进行调控来实现采收率的提高, 已被现场试验证明有效, 且简单易行、成本低廉^[5,6]. 岩石表面润湿性反转被广泛认为是该技术提高原油采收率的主要作用机理^[6~14]. 虽然硅酸盐岩油藏中原始砂岩(主要成分为SiO₂)表面亲水, 但在地层水和原油的长期作用下, 其强极性表面会吸附原油中的极性分

子而变成亲油表面^[15,16]. 对于极性分子在岩石表面的吸附作用, 目前提出的主要机理有范德华力及氢键导致的极性作用、阴/阳离子交换导致的酸/碱性作用和阳离子桥、水桥及配位桥导致的离子桥接作用等^[16~19]. 因此, 低矿化度水驱提高原油采收率的关键机理就是在低浓度离子的作用下使岩石表面吸附的极性分子脱附, 进而把亲油性岩石表面转变为亲水性表面^[10,20~22].

在低矿化度水驱技术中, 润湿性反转的微观机制争议很大, 主要是由于油藏中岩石种类性质、原油组分、地层水组分等条件各不相同, 水驱过程中多种作用错综复杂. 目前提出的主要机制包括双电层膨胀、

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多组分离子交换、pH升高、微粒迁移等^[23-26]。Qi等人^[7]进行了石英-水-原油系统接触角测量,研究了石英表面沥青质甲苯溶液老化时间、沥青质浓度、石英表面的水膜和水膜里的金属离子对表面润湿性的影响,发现水膜里的金属离子能提高岩石表面的油润湿性。Mugele等人^[25]用接触角实验和原子力显微镜(atomic force microscopy, AFM)观察证明, Ca^{2+} 离子在黏土矿物云母上的吸附会导致润湿性转变,并发展了一个基于Derjaguin-Landau-Verwey-Overbeek(DLVO)理论的定量模型来解释这种转变是由表面电荷引起的。随后,Haagh等人^[24]在此基础上进一步发现,随着二价阳离子浓度从一般海水浓度减少到0,白云母的水接触角最多可减少25°,而二价阳离子浓度一定时减小离子强度却对接触角没有影响(离子强度等于溶液中每种离子的质量摩尔浓度乘以该离子的价数的平方所得诸项之和的一半,是衡量溶液中存在离子所产生的电场强度的量度。溶液中离子的浓度越大,离子所带的电荷数目越多,离子强度越大。而离子浓度是指溶液中含某种离子的物质的量与体积之比)。他们认为,在此种情况下影响润湿性的机理是多组分离子交换而非双电层扩散。总之,低矿化度水驱过程中的润湿性反转机制目前存在很多争论,还需要进一步深入地研究。

对于强极性带负电的 SiO_2 表面,质子化后带正电的碱性极性分子很容易静电吸附在其表面,产生碱性作用。同时,含氮原子的极性分子拥有较强的极性,可通过极性作用吸附在 SiO_2 表面。在目前提出的润湿性反转机制中,双电层膨胀机制认为水中离子强度的下降会导致带电岩石和极性分子之间的双电层斥力增加,从而导致极性分子的脱附;其主要针对离子桥接作用和部分极性作用吸附的极性分子。多组分离子交换机制则认为水中带正电的阳离子会置换由碱性作用和离子桥接作用吸附在岩石表面的极性分子。本研究选取原油中常见的胺类物质代表油中碱性极性组分,使用一个仅含一种极性分子的实验模型来排除原油中各种组分和其他作用的影响,研究水中离子对岩石表面极性分子脱附的影响及其对岩石表面润湿性的调控。通过实验测量石英片表面的油-水接触角,获得油中存在碱性极性分子时水中各种无机盐离子浓度对石英片表面润湿性的影响规律,并通过Zeta电位、AFM表征分析了润湿性转变的机制。该研究从复杂的原油-岩石-盐水相互作用体系中提取出单一的成分和作用,对认识低矿化度水驱的基本作用机理有重要意义。

1 实验原理及方法

1.1 实验原理

润湿性是指液体在固体表面铺展的能力或倾向性,由3个界面自由能决定,通常用来度量润湿性强弱的参数为接触角。接触角与界面自由能(界面张力)之间的关系可由杨氏方程给出^[27]:

$$\cos\theta = \frac{\gamma_{\text{so}} - \gamma_{\text{sw}}}{\gamma_{\text{ow}}}, \quad (1)$$

其中 θ 为接触角, γ_{so} 为油-固界面张力, γ_{sw} 为水-固界面张力, γ_{ow} 为油-水界面张力。

研究岩石表面的三相润湿性变化过程时,采用了一个油包水的接触角测量模型,如图1所示。本研究使用石英片(SiO_2)来模拟硅酸盐岩石表面;考虑到石英为强极性表面,在大部分pH下带负电,选用了有强极性且可以质子化带正电的胺类(十八胺)模拟原油中常见的碱性极性分子,将其溶解在模拟油相的癸烷中,配置成十八胺癸烷溶液。将石英片浸没在十八胺癸烷溶液中后,十八胺分子会吸附在石英片表面,从而模拟油藏中碱性极性分子在岩石表面的吸附,使石英片表面变得更亲油。在油中的石英片表面上滴入无机盐溶液液滴,液滴中的离子则会在液-液界面和液-固界面与极性分子发生吸引、反应或置换等,从而再次改变石英片的油-水润湿性。通过拍摄石英片上的液滴图像并测量获得接触角,作为表征石英表面油-水润湿性的参数。

1.2 实验方法

实验需要首先得到干净均一且亲水的石英片表面,

图1 (网络版彩色)实验模型示意图

Figure 1 (Color online) Schematic diagram of experimental model

故需要对石英片进行彻底清洗。先将石英片用自来水和超纯水分别冲洗,然后分别放入无水乙醇和超纯水中超声清洗。随后,将石英片用氮气吹干后浸泡到食人鱼洗液中1 h,以去除石英片表面的各种有机污染物并使其表面充分羟基化。最后,用大量超纯水冲洗干净酸液后用氮气吹干,即得到干净且羟基化的石英片。配置0.1 mmol/L十八胺癸烷溶液及不同浓度的NaCl、KCl、CaCl₂、Na₂SO₄水溶液,并调节水溶液的pH至6.2左右。将清洗干净的石英片浸入配置好的十八胺癸烷溶液中,然后用微量进样器和注射泵在其表面滴入约1 μ L水溶液液滴,使用接触角测量仪(JC-2000,上海中晨数字技术设备有限公司)拍摄液滴侧面图像,并通过轮廓拟合测量其接触角,如图2所示。每片石英片上平行放置4个液滴取平均值,每个液滴等待4 h以上使其达到稳定。

本研究通过测量Zeta电位来表征SiO₂表面的带电性。使用粒径约15 nm的纳米SiO₂,在0.1 mmol/L的十八胺癸烷中超声分散后再离心,去除上清液后加入水溶液,将纳米SiO₂反萃取到水溶液中,再超声分散,即可得到纳米SiO₂水悬浮液。随后立即使用Zetasizer

Nano-ZS(英国马尔文仪器有限公司)测定其Zeta电位。为了直观观察极性分子在石英片表面的吸附和脱附,使用AFM(NT-MDT Solver NEXT型)分别对干净的石英片、十八胺癸烷浸没后取出氮气吹干的石英片,以及浸没在十八胺癸烷中并在表面滴入水液滴后取出吹干的石英片的表面形貌进行扫描。得到3种石英片表面的三维形貌,并求取表面的平均粗糙度及均方根粗糙度。

2 结果与讨论

2.1 离子对接触角的影响

如前所述,清洗干净的石英片表面是亲水的。在纯癸烷环境中,纯水在其表面的接触角约为18°,如图3所示。在癸烷中加入0.1 mmol/L的十八胺后,接触角上升到约42°,即石英片表面润湿性向亲油转变。

当水中加入不同种类和浓度的无机盐时,水溶液接触角均随加入离子浓度的升高而急剧减小(即更亲水),如图4所示。在同浓度下,NaCl溶液和KCl溶液的接触角基本相同,说明Na⁺和K⁺作为单价阳离子效果基本

图2 (网络版彩色)接触角测量系统示意图(a)及测量实物图(b)

Figure 2 (Color online) Schematic diagram (a) and experimental setup (b) of contact angle measurement system

图3 (网络版彩色)石英片在纯癸烷(a)和十八胺癸烷(b)中的接触角

Figure 3 (Color online) Contact angles on clean quartz plates in n-decane (a) and in octadecylaminodecane solution (b)

图4 (网络版彩色)不同溶液接触角随离子浓度的变化。(a) NaCl和KCl溶液接触角对比, 插入图为接触角测量实拍图; (b) NaCl和CaCl₂溶液接触角对比, 插入图为低浓度范围放大; (c) NaCl和Na₂SO₄溶液接触角对比, 插入图为低浓度范围放大

Figure 4 (Color online) Contact angles of different solution droplets vary with the ion concentration. (a) Contact angles of NaCl and KCl solutions, the insertion diagrams are real pictures of contact angle measurements; (b) contact angles of NaCl and CaCl₂ solutions, the insertion diagram shows low concentration range magnification; (c) contact angles of NaCl and Na₂SO₄ solutions, the insertion diagram shows low concentration range magnification

相同。在浓度较低时, CaCl₂溶液接触角减小的速率比NaCl溶液更快, 更早达到稳定值, 在浓度为0.1 mol/L时即基本稳定, 在0.5 mol/L时的略微波动上升是由接触角测量误差引起。在较低浓度下, Na₂SO₄溶液的接触角减小速率很快, 接触角小于NaCl溶液的接触角, 而在较高浓度下, 其接触角与NaCl溶液的接触角接近。最终4种溶液的接触角都趋于15°左右, 接近纯水在纯癸烷中干净石英片表面上的接触角。从以上结果可以看出, 水溶液中的阳离子和阴离子的种类, 尤其是离子价态对接触角均有影响。癸烷中的十八胺在液滴表面吸附并质子化, 得到一个质子带正电, 与液滴中的阴离子相互吸引, 从而促进了其在油水界面上的吸附, 会降低油水界面张力; 而液滴中的阳离子则和质子化后的十八胺同样带正电, 能在带负电的石英表面吸附, 使石英片表面极性减弱, 并与极性分子竞争置换了部分石英表面吸附的十八胺分子。总体而言, 两种作用都会导致吸附了十八胺的石英表面润湿性向亲水转变; 而二价阴阳离子所带电荷量更大, 因此作用更明显。

2.2 实验表征

(i) Zeta电位。石英表面的带电性可通过Zeta电位来反映。如前所述, 干净的石英粉末分散在纯水中具有较高的负电性, Zeta电位为-43 mV左右, 如图5所示。经过十八胺癸烷浸泡再反萃取到水中后, 石英粉末表面转为正电性, Zeta电位约24 mV; 作为对比, 经过纯癸烷浸泡后的石英粉末Zeta电位变化不大, 说明电位转变是由带正电的十八胺在石英表面的吸附引起的, 且十八胺在石英表面发生了多层吸附。将十八胺癸烷浸泡后

图5 (网络版彩色)SiO₂粉末在溶液中的Zeta电位

Figure 5 (Color online) Zeta potential of SiO₂ powder in solution

的石英粉末反萃取到NaCl溶液中, 石英粉末的Zeta电位会减小, 正电性减弱, 甚至在高浓度(1 mol/L)NaCl中迅速重新变为带负电, 说明溶液中离子对极性分子有脱附作用。

(ii) AFM。图6所示为十八胺癸烷浸泡后石英表面形貌三维图、二维图以及线高度分析, 同时给出了干净的石英片、十八胺癸烷浸没后取出氮气吹干的石英片, 以及浸没在十八胺癸烷中并在表面上滴入水液滴后取出吹干的石英片3种情况下10 μm×10 μm范围内的表面平均粗糙度(S_a)和均方根粗糙度(S_q)。可以看出, 干净的石英表面粗糙度较小, 不到1 nm; 经过十八胺癸烷浸泡后, 粗糙度上升, 说明十八胺作为两亲性极性分

图 6 (网络版彩色)石英片表面形貌及粗糙度. (a) 石英表面 $10\ \mu\text{m} \times 10\ \mu\text{m}$ 范围内三维形貌图; (b) 石英表面 $10\ \mu\text{m} \times 10\ \mu\text{m}$ 范围内二维形貌图; (c) 石英表面剖面高度图; (d) 石英表面 $10\ \mu\text{m} \times 10\ \mu\text{m}$ 范围表面平均粗糙度(S_a)和均方根粗糙度(S_q)

Figure 6 (Color online) Surface morphology and roughness of quartz sheets. (a) Three-dimensional morphology of quartz surface within the range of $10\ \mu\text{m} \times 10\ \mu\text{m}$; (b) two-dimensional morphology of quartz surface within the range of $10\ \mu\text{m} \times 10\ \mu\text{m}$; (c) height diagram of quartz sheets; (d) average surface roughness (S_a) and root mean square roughness (S_q) of quartz surface within the range of $10\ \mu\text{m} \times 10\ \mu\text{m}$

图 7 (网络版彩色)润湿性转变机理示意图

Figure 7 (Color online) Schematic diagram of the mechanism of wettability transition

子,其强极性的氨基端与石英片表面作用,使其吸附到了石英片表面;在液滴滴入后,石英片表面的粗糙度有所下降,说明十八胺在离子作用下部分脱附。

2.3 机理讨论

通过以上接触角测量和表征分析,可以将无机盐离子对油-水-石英三相润湿性的影响机理总结如下。如图7所示,当石英片浸没到含有十八胺分子的癸烷溶液中时,十八胺分子会在石英片表面吸附,使石英片表面变得更亲油,因此纯水液滴接触角比在纯癸烷中时大。当水中含有无机盐离子时,水中的阴离子(Cl^- 、 SO_4^{2-})与质子化后带正电的十八胺相互吸引,吸附在油-水界面,导致油-水界面张力下降。同时,水中的阳离子(Na^+ 、 K^+ 、 Ca^{2+})会在带负电的石英片表面吸附,置换已吸附的十八胺分子,导致水-固界面张力降低。这两种作用共同导致了水溶液的接触角下降,石英片变得更亲水。

3 结论

本研究实验测量油-水-固三相接触角,研究了油中存在碱性极性分子十八胺并在石英片表面吸附后,水中 Na^+ 、 K^+ 、 Ca^{2+} 、 Cl^- 、 SO_4^{2-} 等离子对石英片表面润湿性的影响,并通过Zeta电位和AFM表征探究了其机理。实验发现,亲水的石英片表面吸附十八胺后变得更亲油,而盐溶液中的阳离子能通过置换使吸附在石英片表面的十八胺脱附;同时,阴离子能与十八胺吸附在油-水界面从而降低界面张力,也使石英片表面接触角减小。 CaCl_2 、 Na_2SO_4 溶液由于含有二价离子,作用效果比 NaCl 、 KCl 溶液更明显,尤其是在低浓度下。Zeta电位和AFM粗糙度分析均证明了十八胺在石英表面的吸附以及阳离子作用下十八胺的脱附。总之,本研究研究了存在碱性极性分子吸附时水中离子对油-水-石英三相润湿性的影响规律及机理,研究结果对揭示低矿化度水驱的微观机理有重要意义,并有助于低矿化度水驱技术中驱替水溶液离子种类和浓度的设计和调配。

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Summary for “无机盐离子对石英表面油-水润湿性的影响”

Effects of inorganic salt ions on the oil-water wettability of quartz surfaces

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The oil-water wettability in reservoir rock plays an important role in the recovery of crude oil. However, the mechanism of wettability transition on reservoir rock surfaces after low salinity water injection is still controversial. In this paper, the influence of inorganic salt ions in water on the wettability of the rock surfaces with the adsorption of polar molecules and its mechanisms were studied. Amines commonly found in crude oil were selected to represent base polar components in oil, and a highly idealized experimental model was used to exclude the influence of various components and other effects in crude oil. By measuring the oil-water contact angle on the quartz surface, the influence of various salt ion concentration in water on the wettability of quartz in the presence of base polar molecule in oil was obtained, and the mechanism of wettability alteration was analyzed by Zeta potential and AFM measurement. The experiment result showed that, the cleaned surface of quartz was water-wet, when 0.1 mmol/L octadecylamine was added to decane, the contact angle rose to about 42°, that is, the surface of the quartz became more oil-wet. When salts with different kinds and concentrations were added to water, the water contact angles decreased sharply with the increase of the concentration of ions. The contact angles of NaCl solutions and KCl solutions were basically the same at the same concentrations. However, at low concentrations, the CaCl₂ solutions reduced the contact angles more than the NaCl solutions. The contact angles of Na₂SO₄ solutions were smaller than those of NaCl solutions at low concentrations, while the contact angles of Na₂SO₄ solutions were similar to those of NaCl solutions at higher concentrations. When the ion concentrations reached a certain level, the contact angles of different solutions all tended to be about 15°, which indicated that the ions desorbed basically all of the adsorbed polar molecules, leading to the change of wettability of quartz surfaces to be water-wet. The clean quartz powder was highly electronegative when dispersed in pure water. After soaking in octadecylaminodecane, the surface of quartz powder was positively charged. As a contrast, the Zeta potential of the quartz powder after soaking in pure decane changed little, indicating that the potential change was caused by the adsorption of positive octadecylamine on the quartz surface. When the quartz powder soaked with octadecylaminodecane was reversely extracted into NaCl solution, the Zeta potential of the quartz powder would decrease, and the electropositivity would weaken. In high concentration NaCl solution, the Zeta potential of the quartz powder would quickly return to negative charge, indicating the desorption effect of ions in the solution on polar molecules. From the AFM roughness analysis, it can be seen that the surface roughness of clean quartz was small, after soaking in octadecylaminodecane, the roughness increased, indicating that octadecylamine was fully adsorbed on the surface of quartz. After the soaking of droplet, the surface roughness of quartz plate decreased, indicating that the partial desorption of octadecylamine under the effect of ions. Based on the Zeta potential measurement of quartz powder and AFM roughness analysis of quartz surfaces, two mechanisms were proposed: (1) The octadecylamine in decane adsorbed and protonated at oil-water interface, resulting in an electrostatic attraction with the anion, thus reducing the oil-water interfacial tension; (2) the cations in water could be adsorbed on the negatively-charged quartz surfaces, weakening the polarity of quartz surfaces and thus replacing part of adsorbed octadecylamine molecules on the quartz surfaces.

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