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(54) METHOD FOR MEASURING WEIGHT CONCENTRATION OF CLAY IN A SAMPLE OF A POROUS MEDIUM

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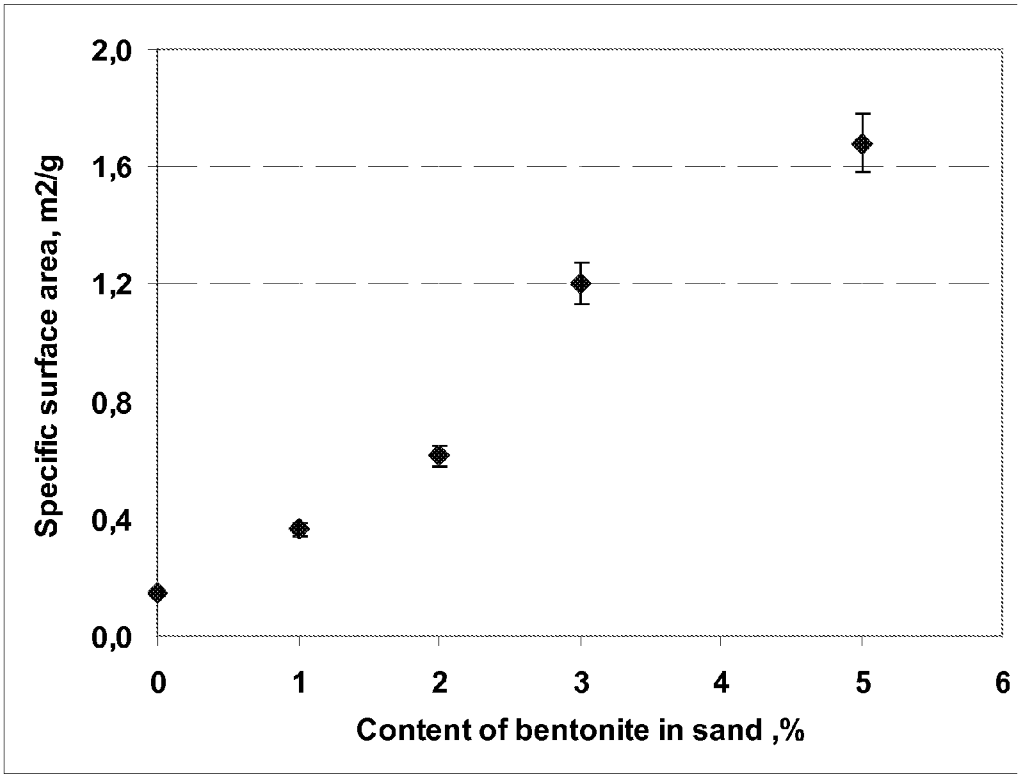
(57) ABSTRACT

For the purpose of determining a weight concentration of a clay material in a porous medium sample, a specific active surface area of the clay material and an initial specific active surface area of the porous sample are measured. A water solution of the clay material is pumped through the sample and a specific active surface area of the sample of the porous medium is measured after the pumping. Then, the weight concentration $n_{гл}$ of the clay material is calculated.



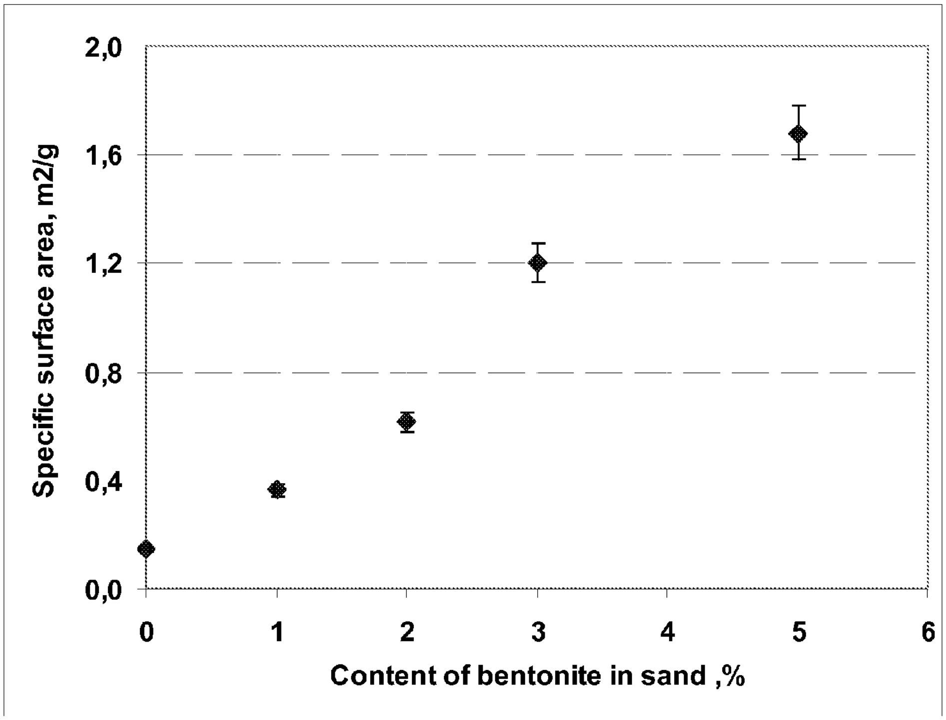

2,0
1,6
1,2
0,8
0,4
0,0
Specific surface area, m2/g
0
1
2
3
4
5
6
Content of bentonite in sand ,%

Fig. 1

METHOD FOR MEASURING WEIGHT CONCENTRATION OF CLAY IN A SAMPLE OF A POROUS MEDIUM

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to Russian Application No. 2012137223 filed Sep. 3, 2012, which is incorporated herein by reference in its entirety.

BACKGROUND

[0002] The problem of formation damage under the impact of a drilling mud (or a flush liquid) is highly important, particularly in the case of long horizontal wells because most horizontal wells are completed without casing, i.e., without production string cementing and perforating.

[0003] Drilling mud is a complex mix of clay, fine particles (from several millimeters to less than one micron), and organic additives (polymers, surfactants, etc.) contained in a "carrying" fluid (a mud base). Such a base fluid could be water, oil, or some synthetic fluid.

[0004] While drilling, mud filtrate together with fine particles and clay contained therein may penetrate the formation near a well bore under excess pressure caising a considerable permeability loss in the affected area. This phenomenon is commonly referred to as "damage to the formation wellbore area" or simply "formation damage."

[0005] A well clean-up technological procedure (gradual transfer to the production mode) can be used to partially wash those components out of the mud damaged zone, which facilitates its permeability buildup. However, some of those components remain captured in the rock pore space (adsorption on the pore surface, capture in pore contractions, etc.), which results in a significant difference between the initial permeability and the permeability built-up following the well clean-up procedure (the built-up permeability would normally represent about 50% to 70% of the initial permeability).

[0006] A laboratory method commonly used to control drilling mud quality is a filtration experiment in which the drilling mud is injected into a core sample with subsequent return (i.e., the displacement of the drilling mud in the sample by the original formation fluid), in which process measures are taken of permeability loss/buildup as a function of the amount of fluid pore volumes (drill mud or formation fluid).

[0007] However, information on the concentrations of clay and other mud components captured in the pore space after the mud has been displaced is important for understanding a mechanism behind formation damage and identifying an appropriate method for improving well productivity factor (minimizing the mud damaged zone). The conventional quality control method of the drilling mud referred to above envisions no such measurements.

[0008] Quantitative analysis of the formation damage mechanism associated with the penetration of argillaceous materials while drilling is of the highest interest in view of the broad use of clay-based drilling mud. For instance, the Russian Federation National Standard ГОСТ 25795-83 recommends bentonite clays for drill mud makeup.

[0009] Weight concentration of drilling mud clay penetrating the pore space is typically small (no more than 1% or 1.5% by weight). Nonetheless, with a high swelling factor of clay and its porosity, even such a small weight concentration results in a significant loss of rock permeability (5 to 20 times down).

[0010] The technical challenge is related to the fact that small weight concentrations of clay in a porous medium are hard to measure because X-ray diffraction and micro-computed tomography methods fail to provide sufficient resolution for weight concentrations of less than 1%.

[0011] U.S. Pat. Nos. 4,540,882 and 5,027,379 disclose methods for determining drill mud penetration depth using X-ray micro-computed tomography of core samples with a contrast agent added thereto. However, the use of a contrast agent dissolved in the "carrying" fluid would prevent determination of penetration and concentrations of clay and other weak-contrast admixtures contained in the drilling mud because the drill mud filtrate penetration depth generally differs from that of the said admixtures.

[0012] U.S. Pat. No. 5,253,719 offers a method for identification of formation damage mechanism through analysing radial-oriented core samples recovered from the well. Core samples are analyzed with the help of various analytical methods identifying the type and extent of formation damage and depth of the mud-damaged zone. The analytical methods used include a X-Ray Diffraction (XRD) method, a Local X-Ray Spectral Analysis, Scanning Electron Microscopy (SEM), Back-Scattering Electron Microscopy, Petrography Analysis, and Optical Microscopy.

SUMMARY

[0013] Various embodiments of the invention provide for measuring small weight concentrations of clay material entering pore space while clay-containing fluid is injected.

[0014] The method for determining weight concentration of clay materials comprises the following processes: measuring an active surface area of a clay material and an initial specific active surface area of a porous sample, pumping a water solution of the clay material through the sample, measuring a specific active surface area of the porous sample after the pumping, and calculating weight concentration of clay material $n_{гл}$ in the porous sample as

$$n_{гл} = \frac{S_{\Sigma}^{уд} - S_{п}^{уд}}{S_{гл}^{уд} - S_{п}^{уд}}$$

where $S_{\Sigma}^{y\delta}$ is the specific active surface area of the porous sample after the pumping of the water solution with clay material, $S_{n}^{y\delta}$ is the initial specific active surface area of the porous sample, and $S_{гл}^{уд}$ is the specific active surface area of the clay material.

[0015] Prior to measuring the specific active surface area of the porous sample and after pumping the clay material water solution through the sample, the sample may be dried.

[0016] Various embodiments of the present invention can be used for nondestructive testing of porous materials. Specifically, the invention can be used for quantitative analysis of property deterioration of oil and gas bearing formations (formation damage) resulting from penetration of clay materials contained in a drill mud.

BRIEF DESCRIPTION OF DRAWINGS

[0017] The invention is illustrated by:

[0018] FIG. **1** shows the dependence of specific surface area of bentonite clay/sand fine mixtures from clay levels (by mass).

DETAILED DESCRIPTION

[0019] The physical basis of the above method is that additives increase within a specific active surface area of a porous sample with the buildup of clay levels (by mass) in its pore space:

$$S_\Sigma^{y\delta} m_\Sigma = S_n^{y\delta} m_n + S_{\text{гл}}^{\text{уд}}\, m_{\text{гл}} \tag{1}$$

where m_n and $S_n^{y\delta}$ are mass and specific active surface area of a porous sample before exposure to an argillaceous material water solution; $m_{\text{гл}}$ and $S_{\text{гл}}^{\text{уд}}$ are mass and specific active surface area of clay used for making up the argillaceous material water solution; m_Σ and $S_\Sigma^{y\delta}$ are mass and specific active surface areas of a porous sample after exposure to an argillaceous material water solution.

[0020] Combining equation (1) with the mass balance of:

$$m_\Sigma = m_n + m_{\text{гл}}, \qquad (2),$$

weight concentration of clay $n_{\text{гл}}$ may be obtained in the sample with known $S_n^{y\delta}$, $S_{\text{гл}}^{\text{уд}}$ and $S_\Sigma^{y\delta}$:

$$n_{\text{гл}} = \frac{S_\Sigma^{\text{уд}} - S_n^{\text{уд}}}{S_{\text{гл}}^{\text{уд}} - S_n^{\text{уд}}}, \text{ where } n_{\text{гл}} = \frac{m_{\text{гл}}}{m_\Sigma}. \tag{3}$$

[0021] Considering that clay features high specific active surface area, the presence of even small levels of clay in pores results in a sharp increase of specific active surface area of the porous sample.

[0022] A series of special metrological experiments were conducted to measure specific active surface area of bentonite clay/sand fines mixtures to verify that additives within a specific active surface area of the mixture increases with increasing levels of clay (by mass).

[0023] Using a conventional nitrogen sorption method based on the Brunauer-Emmett-Teller (BET) Theory (See for instance, O. M. Poltorak, Thermodynamics in Physical Chemistry, M, Higher School, 1991, Pages 172-176), specific active surface area of the bentonite clay was measured, as well as specific active surface area of sand fines. A mixture was made up of sand fines and bentonite clay with a preset weight concentration of clay. Using the same nitrogen sorption method (BET), specific active surface area was measured of the said bentonite clay/sand fines mixture. The measurements were repeated for bentonite clay/sand fines mixtures with various weight concentrations of clay. The experimentally obtained values of specific active surface of bentonite clay/sand fines mixtures with various clay levels (by mass) are shown in FIG. **1**. The experimental data allow for good approximation by linear dependence, which confirms the theory that additives increase within the specific active surface area.

[0024] In an example, the invention was used to determine bentonite clay weight concentration penetrating a porous sample, while pumping 1% solution of said clay through that sample.

[0025] The conventional nitrogen sorption method based on the BET theory was used to measure specific active surface area of bentonite clay: $S_{\text{гл}}^{\text{уд}} = \mathbf{32.85}\ \text{m}^2/\text{г}$ and 1% water solution of that clay was made up (which is close to clay concentration in actual drilling muds) with a sodium salt additive (18 g/l).

[0026] The conventional nitrogen sorption method based on the BET theory was used to measure original specific active surface area of a porous material sample prior to pumping a clay-containing solution through the sample:

$$S_n^{y\delta} = 1.27\ \text{m}^2/\text{г}$$

[0027] After that, the 1% clay solution made up for the purpose was pumped through that porous sample. A total of seven pore volumes were pumped through (pumped solution volume/sample pore space volume ratio), following which filtration actually stopped in view of considerable permeability loss. At that point, the experiment stopped.

[0028] The porous sample was dried up and a specific active surface area of the same porous sample was measured by the conventional nitrogen sorption method based on the BET theory after seven pore volumes of 1% clay solution had been pumped through the sample:

$$S_\Sigma^{y\delta} = 1.38\ \text{m}^2/\text{г}$$

[0029] Using equation (3) and the values of specific active surface areas of clay measured at earlier stages as well as those of the porous sample before and after pumping the clay-containing solution, weight concentration was calculated of the bentonite clay which penetrated into the porous sample:

$$n_{\text{гл}} \approx 0.35\%$$

1. A method for determining a weight concentration of a clay material in a sample of a porous medium, the method comprising:

measuring a surface area of the clay material and an initial surface area of the sample of the porous medium,

pumping a water solution of the clay material through the sample of the porous medium,

measuring a surface area of the sample of the porous medium after the pumping, and

calculating the weight concentration $n_{\text{гл}}$ the clay material in the sample of the porous medium as

$$n_{\text{гл}} = \frac{S_\Sigma^{\text{уд}} - S_n^{\text{уд}}}{S_{\text{гл}}^{\text{уд}} - S_n^{\text{уд}}}$$

where $S_\Sigma^{y\delta}$ is the surface area of the sample of the porous medium after the pumping of the water solution of the clay material, $S_n^{y\delta}$ is the initial surface area of the sample of the porous medium, and $S_{\text{гл}}^{\text{уд}}$ is the surface area of the clay material.

2. The method of claim **1**, wherein the sample of the porous medium is dried prior to measuring its surface area after pumping the water solution of the clay material through the sample.

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