

Description

This invention relates to centrifuges and more particularly, relates to automated centrifuges.

The oil industry has developed several methods for measuring capillary pressure and relative permeability of reservoir core samples using centrifuges. Capillary pressure and relative permeability are important properties for describing the flow of fluids in porous media and are generally needed in the reservoir engineering of an oil field. These properties help the reservoir engineer determine, for example, the productivity of a reservoir, the total reserves, and the likelihood of success for various oil recovery processes, such as water flooding or carbon dioxide flooding.

One of the methods for measuring capillary pressure is the centrifuge method of Hassler and Brunner (Hassler, G.L. and Brunner, E., "Measurement of Capillary Pressures in Small Core Samples", Trans. AIME 1945, Vol. 160, p. 114-123). Similarly, a method for measuring relative permeability is the centrifuge method of Hagoort (Hagoort, J., "Oil Recovery by Gravity Drainage", Soc. Pet. Eng. J., June 1980, Vol. 28, p. 139-150).

These methods have the advantage of much greater speed compared to other methods for measurement and are amenable to automation. For both centrifuge methods, the core samples are mounted in special holders having glass collection tubes to allow for monitoring the production of fluid from the core samples. The cores are centrifuged and the effluent fluids from the samples are collected in the tubes. A strobed light source is used to determine the amounts of fluids collecting in the glass collection tubes.

Measuring the capillary pressure with the method of Hassler and Brunner requires increasing the speed of the centrifuge in "steps" or increments and measuring the amount of fluid produced from the core sample when all flow has ceased for that step (i.e. centrifuge speed) before increasing the centrifuge speed to the next "step". Measuring the relative permeability with the method of Hagoort requires running the centrifuge at a single speed, which is high enough to overwhelm capillary pressure effects, and measuring the amount of fluid produced from the core sample as a function of time.

In general, however, the capillary pressure or relative permeability curve determined by the prior art in a centrifuge experiment use effluent data alone. Since no measurements are made of the fluid saturations inside the core, various assumptions must be made concerning boundary conditions, uniformity of the displacement of the fluid, and homogeneity of the core. These assumptions may not always be valid, leading to inaccurate and unreliable results. However, even if these assumptions are valid, capillary pressure and relative permeability influence the measurement of each other.

The Hassler and Brunner method for measuring capillary pressure is confined to a drainage mode of flow for a water-wet core initially filled with a wetting fluid which is then invaded by a non-wetting fluid, i.e., oil invading a water-wet core. The method of Hassler and Brunner is not useful when a wetting fluid invades a water-wet core containing a non-wetting fluid as the equilibrium level of production of the non-wetting fluid is dependent upon imbibition and not centrifuge speed. However, such measurements are needed in order to design waterflood recovery methods where the invading fluid is wetting.

Further, EP-A 0 106 398 discloses a method for measuring the distribution of fluids in a sample, or for measuring the linear attenuation coefficient of a fluid in a sample, or for determining the relative permeability curve of a sample, or for determining the capillary pressure curve for a sample, comprising the steps of centrifuging said sample; and periodically imaging said sample in a plurality of points during centrifuging.

This reference also describes an apparatus for measuring the distribution of fluids in a sample, comprising: a centrifuge having a predetermined number of sample holders for containing said sample and capable of operating at plurality of speeds; a source of electromagnetic energy positioned to radiate onto at least one preselected holder; and a detector array positioned to detect electromagnetic energy transmitted through said at least one preselected holder.

Further, US-A 3 599 002 describes a method and an apparatus for measuring the distribution of fluids in a centrifuged sample. According to this reference, two preselected electromagnetic energies may be used.

The known centrifuges, however, are not suitable to measure resistivity of the sample during centrifuging.

These and other limitations and disadvantages of the prior art are overcome by the present invention however, and it is an object of the invention to provide an improved method and apparatus for centrifuging core specimens that are capable of measuring resistivity of the sample during centrifuging.

The invention therefore provides a method for measuring the distribution of fluids in a porous sample, comprising the steps of:
centrifuging said sample;
periodically imaging said sample in a plurality of points during centrifuging; characterized by the step of measuring the resistivity of said sample during said centrifuging.

The invention also provides an apparatus for measuring the distribution of fluids in a sample, comprising:
a centrifuge having a predetermined number of sample holders for containing said sample and capable of operating at plurality of speeds;
a source of electromagnetic energy positioned to radiate onto at least one preselected holder;

a detector array positioned to detect electromagnetic energy transmitted through said at least one preselected holder;
 means for periodically imaging of the said sample in a plurality of points during centrifuging;
 characterized in that the sample holders are adapted to allow resistivity measurements during the centrifuging process.

These and other features and objects of the present invention will become apparent from the following detailed description wherein reference is made to the Figures in the accompanying drawings, in which:

Fig. 1 shows a side view, partially in cross section, of an X-ray centrifuge based upon known principles;

Fig. 1A shows an enlarged cross-section of a portion of the apparatus depicted in Fig. 1;

Fig. 2 shows a schematic block diagram of one embodiment based upon known principles;

Fig. 3 shows a block diagram of an alternate embodiment of an apparatus based upon known principles;

Fig. 4 shows an enlarged cross-section of a portion of the apparatus depicted in Fig. 1;

Fig. 5 shows typical X-ray tube spectrum for two different peak acceleration voltages;

Fig. 6 shows the mean X-ray energy of a typical X-ray tube spectrum as a function of peak acceleration voltage;

Fig. 7 shows linear attenuation coefficient versus energy for selected dopants;

Fig. 8 shows an alternate embodiment of an apparatus based upon known principles;

Fig. 9 shows an embodiment of the apparatus of the present invention;

Fig. 10A and 10B show the apparatus of the present invention employing the embodiment depicted in Fig. 9;

Fig. 11 shows another alternate embodiment of an apparatus based upon known principles; and

Fig. 12 shows yet another alternate embodiment of an apparatus based upon known principles.

Referring now to Fig. 1, there may be seen a side view, partially in cross-section, of the apparatus 2 based upon known principles. More particularly, there may be seen a conventional centrifuge 10. This conventional centrifuge 10 consists of a motor 12, which drives the shaft 14. The shaft 14 is connected to a rotor 16, which holds sample holders 18 by any suitable means, such as, for example but not limited to the pins 20. The sample holders 18, in turn, may contain the samples (not shown) to be centrifuged. The centrifuge 10 may also contain a suitable synchronizing means 22 on the shaft 14.

A source of electromagnetic energy 24, such as for example, but not limited to an X-ray tube is positioned adjacent the centrifuge 10 so that the radiation or X-rays emitted therefrom may be collimated by a lead collimator 25 and then impinge on the sample holder 18, which may contain a sample (not shown). A flash X-ray tube emits a burst of X-rays in approximately 30 nanoseconds with an intensity of about 60 milliroentgens at 1 metre. This short duration burst of X-rays (which is 100 times faster than optical strobes) is sufficiently fast that the motion of the centrifuge is effectively frozen, or stopped, during the time the X-ray energy passes through a core sample and/or holder 18. Such a quick burst of X-ray or other radiation allows for effectively "freezing" the motion of the centrifuge. For example, for a centrifuge arm of about 15 cm at a rotational speed of about 20,000 rpm, the sample movement is about 10 μ m during a 30 nanoseconds burst of radiation. For rotational speeds greater than this, alternate embodiments of the present invention, as described later herein (see Fig. 11), may be employed; these alternate embodiments may also be employed for slower rotational speeds.

A detector means 26, which may be for example, but not limited to a fluorescent screen, may be positioned on the opposite side of the sample holder 18 from the X-ray tube 24 so that at least a portion of the X-rays from the X-ray tube 24 that pass through the sample holder 18 and any sample will impinge upon the detector means 26. The detector means 26 may be situated as close to the sample holder 18 as possible, to obtain the sharpest image.

Alternatively, the detector means 26 may be located to the side of sample holder 18 and moved to its detecting position only after the centrifuge has begun rotation and the holder 18 is in its "rotating" horizontal position, as depicted in Fig. 1. This allows the detector 26 to be much closer to the sample and to minimize any radiation beam dispersion. In a similar manner and for an alternate embodiment of the present invention, the radiation source may be similarly mounted in such a movable fashion and the detector 26 may be fixed above and immediately adjacent the holder 18 in its horizontal position. Alternatively, the radiation source 24 may be permanently mounted below the sample holders 18, and the detector 26 mounted above the rotating position of holders 18, as described later herein (see Fig. 12).

The fluorescent screen material is chosen to have high quantum efficiency for the energy of the incident radiation or X-ray spectrum. In addition, the fluorescent screen must be sufficiently thick to ensure that a substantial portion of the available radiation or X-ray energy is converted to optical energy. In addition, the phosphor is chosen so that the fluorescence from the screen has decayed before the next radiation or X-ray burst is initiated which can be, for example but not limited to, 1 second later than the preceding burst.

Referring now to Fig. 1A, the detector means 26 is depicted in more detail. For this detector means 26, a fluorescent screen 27 may be employed to detect radiation and emit light as a result of this detection. The light output from the fluorescent screen 27 may be focused by the lens 28 or fibre optic cables (not

shown) onto an image intensifier 29. The region from lens to image intensifier is shrouded by the light cover 30 to prevent stray light from entering the assembly. The function of the image intensifier tube is to provide suitable amplification (of up to 100,000 times) so that only a single radiation or X-ray pulse or burst is necessary to obtain a high quality image. The amplified output from the image intensifier tube may be detected by film, a television camera, or a solid state array camera 31. The solid state array camera may contain, for example, a 256 by 256 square array of light sensitive photodiodes, or some other type of solid state array, each of which is 25 micrometres square and which are spaced 25 micrometres apart, mounted inside a camera such as the camera discussed hereinabove. The solid state array 31 can, in general, have an M by N array of photodiodes. Alternatively, a direct detector of radiation or X-rays in an array arrangement may be employed as detector means 26 to directly detect X-rays, or other types of electromagnetic radiation, without the use of a screen or image intensifier.

In this manner the position of fluids within the sample may be determined at a plurality of points (projections into a two-dimensional detector array). This plurality of points allows for very precise location of fluids and their saturations within the sample, as well as precise detection of movement of these saturations in the sample. The output of the detector may be directed to various storage or recording devices.

Alternatively, another source of pulsed X-rays, such as, for example, but not limited to synchrotron radiation from a cyclotron (not shown), may be employed to irradiate the sample and sample holder. Another alternative embodiment may employ a constant radiation source, (such as, for example, but not limited to a rotating anode X-ray tube) and employ a pulsed or strobed detector to only look at the detector output when the holder and sample is between the radiation source and detector.

Referring now to Fig. 2, there may be seen a schematic block diagram of one embodiment of the apparatus based upon known principles. More particularly, a computer/controller 52 is depicted interconnected with a speed controller 54, a strobe controller 56, and an analog-to-digital converter 58. The strobe controller 56 provides the strobe pulse to the X-ray tube power supply 60 which energizes or "fires" the X-ray tube 24, which may be a flash X-ray tube. The X-ray are depicted as arrows emanating from collimator 25 and passing through the sample holder 18 (containing a sample) before being detected by the detector 26. The output of the detector 26 is supplied to the analog-to-digital converter 58, which digitizes the detector's output and then supplies the digitized output to the computer/controller 52. There is also an interconnection between the speed controller 54 and the strobe controller 56, which provides for synchronization between these two controllers such that the X-ray tube is "fired" when the appropriate holder 18 and sample is located beneath the X-ray tube. This is accomplished by signals from the synchronizing means 22.

The synchronizing means 22 needs to be capable of imaging the sample at the same position independent of the speed of operation of the centrifuge. However, at the highest operating speeds (approximately 20,000 rpm), the fixed time delays in the strobe controller and X-ray power supply may cause the X-ray tube to fire too late, resulting in an image of the sample shifted relative to the imaging array. The synchronizing means may be calibrated at each speed to cause the strobe controller to fire earlier as the centrifuge speed is increased, to avoid this shifting of the sample relative to the array.

The computer/controller 52 may be a microcomputer which performs the control functions described hereinbelow as well as providing data storage and real time processing of the output data provided by the detector 26. Alternatively, the various control functions may be performed by a separate controller circuit which directs the output from the detector 26 to a microcomputer which can then perform the data storage and initial processing.

The speed controller 54 controls the speed at which the rotor 16 of centrifuge 10 of Fig. 1 is rotated by controlling the speed of the motor 12. The computer/controller 52 provides a signal to the speed controller 54 which indicates the desired rotor speed. Although the rotor may be run at one speed for some preselected period of time and then the speed of the rotor increased in predetermined steps with a fixed period of time allocated at each step, the computer/controller 52 may control speed in any manner desired. The strobe controller 56 provides a predetermined number of strobe pulses to the X-ray tube power supply 60 in response to a control signal from the computer/controller 52.

The solid state camera 31 (see Fig. 1A) may consist of any suitable solid state array, such as for example, but not limited to a photodiode array and associated circuitry mounted in a suitable camera, as is presently preferred. Each of the photodiodes in the two-dimensional array may be connected in parallel with a capacitor which is charged to a predetermined saturation charge. This charge is leaked off by current generated when light impinges on the photodiode. The remaining charge and the voltage across the capacitor is proportional to the amount of light striking the respective photodiode during a fixed exposure time.

The photodiode array is scanned by a digital shift register which sequentially causes the voltage across the respective capacitors in each row, one row at a time, to be sent to a common output line. The capacitor is then refreshed to its original saturation charge and the shift register moves on to the next photodiode in the array. The photodiodes are constantly being scanned and refreshed at a frequency determined by the computer/controller 52. When the computer/controller 52 determines that it is time to collect a data sample, it waits until the last photodiode in the array has been scanned and refreshed. It then stops the scanning process and signals the strobe controller 56 to pulse the X-ray power supply.

The computer/controller 52 then waits a predetermined period of time after the X-ray pulse to ensure collection of sufficient light output from the phosphor screen 27 and it then signals the solid state camera 31 to restart the scanning process. It should be noted that the only light that is seen by the photodiode array is the light provided by the fluorescence of the phosphor screen 27, as intensified by the image intensifier 29.

The output signal provided by each photodiode and capacitor combination is provided to the common output line by the shift register. The common output line is connected to the analog-to-digital converter 58, which is preferably a part of the computer/controller 52, although it may be separate from the computer/controller 52. Analog voltages are converted to equivalent digital signals by the analog-to-digital converter 58, which may then be stored and/or processed by the computer/controller 52. This digitized output may be directed to a cathode ray tube monitor or other visual output devices, such as pen recorders for each row of the array, immediately after the photodiode array has been scanned to provide a visual display of the data.

For a constant radiation source, or constant X-ray source, the outputs of the detector may be ignored until the preselected holder is positioned between the source and detector, as noted by an appropriate signal from the computer/controller 52. Then, the detector signal is treated as noted hereinbefore.

Referring now to Fig. 3, there may be seen a simplified block diagram of an alternate embodiment of the apparatus based upon known principles. The speed controller 54, the strobe controller 56 and the solid state camera 31 have been represented. A cathode ray tube monitor 74 is shown appropriately connected to the computer/controller 52 as described hereinbefore. The cathode ray tube monitor is the monitor on which the digitized output may be displayed. Further, the digitized image data may be stored on an appropriate storage media 72. The computer/controller 52 may also be connected by a high speed serial or parallel port to a remote processing computer 70. The computer 70 may then calculate fluid saturations, generate saturation images and may provide animated movies of saturation change during fluid displacement processes. The calculation of fluid saturations is based upon intensity information obtained as described hereinabove.

Further, the computer 70 may be linked to other high-speed computers (not shown). Such a high-speed computer may be employed to calculate numerical simulations of fluid saturations (or other parameters) and their changes for the operating conditions of the centrifuge for various assumed sample characteristics. These assumed sample characteristics may be changed until the numerical simulation gives good agreement to the actual centrifuge data. Then the simulation and centrifuge results may be displayed together, in any suitable format, such as for example, but not limited to, animated movies, video tape, or colour cathode ray tube displays.

However, to determine fluid saturations, intensity images must be obtained with the core fully saturated with each fluid whose saturation is to be measured. These intensity images (fully saturated core) are used with those from the centrifuging process to compute fluid saturations (based upon Beer's Law, as described later herein) and then to generate saturation images from which animated movies may be made.

It should be noted that the rate at which images can be collected will be determined by the rate at which the analog voltages can be clocked out of the photodiode array and digitized. For an image each second from a 256 by 256 photodiode array with 8 bit resolution, the clocking and digitization rates must exceed $5.25 \times 10^5/s$ (i.e. approximately one-half a megahertz). This can be achieved with standard analog-to-digital electronics as is well known in the art.

Referring now to Fig. 4, there may be seen a side view, partially in cross-section, of one embodiment of a sample holder 18. This sample holder 18 is adapted for holding a core sample 32 from a down hole core of an oil well. This sample 32 and the sample holder 18 is served by way of illustration and not by limitation since the apparatus of the present invention can also be employed to analyze various porous solids, mixtures and fluids, for example, but not limited to soils, concrete or human blood. The sample holder 18 comprises a core housing 36 which has a chamber 38 that is suitably sized to accommodate core sample 32. The core housing 36 is constructed of suitable material to be transparent to the radiation employed, for example, aluminium may be employed for X-ray radiation. The core housing 36 is provided with a lid 34 to facilitate loading and unloading the sample. The lid 34 may have a pressurization port 35 and a valve 37 to pressurize the chamber either with an overburden gas or a high pressure displacement gas such as carbon dioxide. A spacer ring 40 is positioned around the core sample 32 and a support ring 42 is positioned between the core sample 32 and the bottom of sample holder 18.

The sample holder and sample may have a precision bore and a square cross section which results in uniform radiation or X-ray path lengths over the entire core sample. The spacer ring 40 can be made of any suitable resilient material which is suitably radiation or X-ray transparent. The support ring 42 has an open volume that allows any ejected fluids to accumulate and be measured after the centrifuging process. A separate collection chamber (not shown) may also be located at the bottom of the sample holder 18, as is known in the art, so that accumulated fluids may also be measured during the centrifuge process.

A reference standard 44 may be included to provide a suitable radiation absorption standard in the image which allows normalization for flux variations. The reference standard 44 may be any suitable material with a known linear attenuation coefficient and good transparency to the radiation employed, such as for example, but not limited to fused quartz for X-ray radiation.

The basic quantity measured by this invention is the linear attenuation coefficient μ . This is defined from Beer's Law, $I/I_0 = \exp(-\mu x)$, where I_0 is the incident radiation intensity and I is the intensity remaining after passing through a thickness, x , of material having a linear attenuation coefficient, μ . Beer's Law assumes a narrow radiation beam and monochromatic (single energy) radiation. An X-ray tube does not emit monochromatic X-rays, however, but emits X-rays having a spectrum of energies $D(E)$, where $D(E)$ is the relative photon energy between the energies E and $E + dE$. The mean X-ray energy, $\bar{E}$, of the spectrum $D(E)$ is defined as:

$$\bar{E} = \int_0^{E_{\max}} D(E) E dE / \int_0^{E_{\max}} D(E) dE$$

where $E_{\max}$ is the peak acceleration potential. With no absorbing object (i.e. no sample or holder) between the source and detector, the detector output, V_0 is:

$$V_0 = K \int_0^{E_{\max}} D(E) dE$$

where K is the detector proportionality constant. However, after radiation passes through an absorbing object, the detector output V is:

$$V = K \int_0^{E_{\max}} D(E) \exp(-\int_1 \mu(r, E) dl) dE$$

where the inner integral is taken over the path length l between source and detector, $\mu(r, E)$ is the spatial and energy dependent linear attenuation coefficient, and K is the detector proportionality constant. Thus, by comparing V/V_0 , the path integral of μ may be determined through the material. Since the thickness of the core is known, μ may be determined for the mean energy of the X-ray beam.

Referring now to Fig. 5, there may be seen a depiction of X-ray tube spectra for two different peak acceleration voltages. The horizontal axis represents energy (in KeV) and the vertical axis represents relative energy. More particularly, Fig. 5 plots the energy spectrum generated by a typical tungsten target X-ray tube with two different peak acceleration potentials of 90 kilovolts and 150 kilovolts. This Figure demonstrates that the mean energy of an X-ray tube emission spectra may be changed by employing different acceleration potentials, as discussed later herein.

Referring now to Fig. 6, there may be seen a depiction of the mean X-ray energy (vertical axis) of a typical tungsten target X-ray tube spectrum as a function of peak acceleration voltage (horizontal axis). More particularly, Fig. 6 shows the mean beam energies emitted by an X-ray tube as a function of peak accelerating potential with and without the filtration provided by 1.5 inches of aluminium and 2 inches of sandstone with 20 percent porosity. The line A represents detected spectra, whereas the line B represents emitted spectra. Thus, Fig. 6 illustrates a representative change in the energy spectrum emitted by an X-ray tube caused by passage through a sample and sample holder. Similar curves may be generated for other sources of electromagnetic radiation. For two-phase systems, X-ray absorption measurements need only be made at one X-ray energy to determine linear attenuation coefficients in a plurality of points. These coefficients may then be used to determine fluid saturations at those points. As an example, consider an oil/water mixture at a point, for which two equations may be written:

$$\begin{aligned} \mu &= \mu_w S_w + \mu_o S_o \text{ and} \\ 1 &= S_w + S_o \end{aligned}$$

which may be combined, resulting in one equation:

$$\mu = (\mu_w - \mu_o) S_w + \mu_o, \text{ or } S_w = \frac{\mu - \mu_o}{\mu_w - \mu_o}$$

where μ is the linear attenuation coefficient actually measured, μ_w and μ_o are the attenuation coefficients of water and oil for this sample, respectively (which have already been measured) and S_w and S_o are the calculated saturations of water and oil, respectively. For a two-phase system, one of the phases may be doped with a highly absorbent material to increase the difference between μ_w and μ_o .

For three-phase systems, at least two independent measurements need to be made to determine the

saturations. If two X-ray absorption measurements are made at X-ray energies E_1 and E_2 , two equations result:

$$\mu_1 = \mu_{w1} S_w + \mu_{o1} S_o + \mu_{g1} S_g$$

$$\mu_2 = \mu_{w2} S_w + \mu_{o2} S_o + \mu_{g2} S_g$$

A third equation is also available: $1 = S_w + S_o + S_g$

Here, μ_1 and μ_2 are the linear attenuation coefficients of the sample with multiple phases present at E_1 and E_2 , respectively. The terms μ_w and μ_o and μ_g are the attenuation coefficients of the sample fully saturated, respectively, with each one of the three phases, which have been taken for this example to be water, oil and gas. S_w , S_o , S_g are the respective saturations of these three phases. The third equation states that the pore volume of the sample is completely filled by some combination of these three phases.

These three equations must be linearly independent in order for there to be a unique solution for the three saturations. The ability to obtain two independent measurements by using two different X-ray energies is based on the fact that X-ray attenuation may depend on density as well as on chemical composition. For energies above about 100 KeV X-rays interact with matter predominantly by Compton scattering, which is dependent upon electron density. For X-ray energies well below 100 KeV, photoelectric absorption becomes dominant. Photoelectric absorption is strongly dependent upon atomic number. The heavier elements have greater photoelectric contribution which increases rapidly as the X-ray energy is lowered. In addition, many of the heavier elements have K-absorption edges near the energy range of about 60 to about 90 KeV.

Referring now to Fig. 7, there may be seen a depiction of the linear attenuation coefficient (vertical axis) versus energy (horizontal axis) for two selected dopants. The line A represents $(Na)_2(WO_4)$, whereas the line B represents iodododecane. The line C represents gas. These two dopants may be placed into the various phases which are in a core sample and used to uniquely identify which phase is absorbing which X-ray energy. The best-conditioned solution (i.e., least sensitive to measurement error) to the foregoing three equations is obtained if two dopants are used, one with a K-absorption edge above the energy E_2 and another whose absorption is continuous in this energy range. One dopant can be used in each phase and the concentrations are adjusted for maximum sensitivity. An example of dopant and X-ray energy combination that gives good results are $E_1 = 150$ Kvp (92 KeV mean), $E_2 = 90$ Kvp (64 KeV mean), where a water phase is doped with one molar Na_2WO_4 and an oil phase is doped with 20 %wt iodododecane. The K-absorption edge for tungsten is about 69.5 KeV; the two energies, E_1 and E_2 noted hereinbefore, have been selected with one energy just below this K-absorption edge. The 69.5 KeV k-absorption edge is located at approximately the position D of Fig. 7. Alternatively, only one phase may need to be doped. For example, if only the oil phase is doped with 20 %wt iodododecane and the same X-ray energies are utilized, good results may still be obtained.

The simultaneous, unique solution to the foregoing three equations is best-conditioned when the radiation at one energy is absorbed primarily by the water phase and the radiation at the other energy is absorbed primarily by the oil phase. In addition, the average absorption levels should be approximately the same at both of these energies. The sensitivity improves with increasing energy spread between the two X-ray energies. However, the low energy scan must still have a high enough X-ray energy to ensure the beam penetrates the sample holder and core without drastic attenuation.

The dual energies needed for three-phase imaging may be achieved by: (1) changing the peak acceleration voltage applied to one X-ray tube, or (2) employing appropriate filters placed in the beam, or (3) employing two X-ray tubes at different voltages, or (4) employing a dual energy detector, or (5) by a combination of any of these methods.

Referring now to Fig. 8, there may be seen a dual energy detection system. Fig. 8 depicts an embodiment that uses a single X-ray tube 24 with a single peak acceleration voltage but having dual energy detectors 26a and 26b. Fig. 8 shows an arrangement of dual energy detectors which utilize a low energy phosphor screen 27a such as calcium difluoride and a high energy screen 27b such as sodium iodide, separated by an X-ray filter 70 which passes only high energy X-rays. Photons emitted by the two screens are detected by two separate cameras labeled as the "low energy camera", 31a and the "high energy camera", 31b.

Alternatively, more dopants may be employed if more than three fluids are present. If more dopants are employed more X-ray energies are also required to provide multiple fluids, as noted hereinbefore.

From the saturations images obtained during the centrifuging process, capillary pressure or relative permeability curves may be rapidly calculated. As a simple example, a sample saturated with water with a density of ρ_w is placed in the centrifuge which is filled with oil with a density ρ_o . The sample is spun at an angular velocity Ω until equilibrium is reached, i.e. no more fluid is produced from the sample. Taking the axis of the centrifuge as the origin, and the axis perpendicular to the axis of the centrifuge as the r axis (directed toward the axis of the centrifuge), the capillary pressure p_c at any location r , spaced from the origin, is $p_c = (\rho_w - \rho_o) \Omega^2 (R^2 - r^2)$, where R is the distance from the centrifuge axis to the end of the sample most distant from the axis; this equation is similar to one derived by Hassler and Brunner for their centrifuge method. The oil and water saturations at that capillary pressure are obtained from the satura-

tion image at the same location. The centrifuging is repeated at different rotational speeds and the capillary pressure curve for each region of the sample is thus determined.

Similarly, the relative permeability is obtained from two sequential images at the same location separated by a time t . If the core is spun at high angular velocity for early times, then capillary pressure effects are small, compared to the relative permeability. Thus, capillary pressure may be neglected. If the invading phase is assumed to have infinite mobility, the water relative permeability K_w at a location r is:

$$K_w = \frac{\mu_w}{K \Omega^2 (\rho_w - \rho_o) r} \int_{r_{in}}^r \phi \frac{\partial S_w}{\partial t} dr$$

where the water saturation is S_w , ϕ is the porosity, K is the permeability, μ_w is the water viscosity, and r_{in} is the radial distance to the inlet face. This equation is similar to one derived by Hagoort for his centrifuge method. As S_w changes during the centrifuging the relative permeability curve for water can be determined at each location of the sample.

These simple examples are intended to demonstrate that either capillary pressure or relative permeability can be determined at every location throughout the sample using the information available from this invention.

Referring now to Fig. 9, there may be seen a sample holder 89 for the X-ray centrifuge of the present invention. This sample holder 89 is adapted for use in this invention to also allow for resistivity measurements with or without overburden pressure during the centrifuging process. This sample holder contains a plurality of electrodes 90 between which the resistivity is measured, mounted on both ends of sample 32. The electrodes 90 may consist of platinized platinum screen to minimize electrode polarization as is well known in the art.

An overburden sleeve 92 separates the samples 32 from the overburden fluid and also forces electric current to flow only through the sample. The overburden sleeve can be any non-conducting flexible material such as shrink-fit Teflon, rubber etc. which makes good contact with the sample so that current cannot flow around the exterior surface of the sample. The sample is fitted with an endpiece 93 through which the resistivity lead 94 passes. The sample holder container 98 is fitted with a cap 99 secured by cap bolts 97. An O-ring 100 establishes the high pressure seal between the cap 99 and the container 98. A pressure-tight seal 96 is provided in the cap 99 for the resistivity lead 94. The container 98 is constructed of appropriate material so as to be effectively transparent to the selected radiation wavelength used to image the sample, such as aluminium for X-ray wavelengths. An overburden valve 101 is used for applying preselected pressure to the overburden fluid before centrifuging. Sample pressure valve 102 is used for applying a preselected pore pressure to the sample before centrifuging. The holder may also have suitable temperature and/or pressure measuring means thereon or therein to allow for measuring the temperature and/or pressure of the sample.

Referring now to Fig. 10A and 10B, there may be seen a simplified representation of the centrifuge employing the resistivity holder of Fig. 9. More particularly, Fig. 10A shows the electrical connection between the sample holder of Fig. 9 and the resistivity monitoring apparatus. The resistivity lead 94 of Fig. 9 may connect to a sliding brush 110 on one of the rotary slip-rings 111; alternatively, the lead 94 may be connected electrically to the bottom of one of the slip-rings 111. Electrical ground may be established through one electrode and aluminium fluid collection container 98 to the metal rotor and shaft. The resistivity lead 94 and ground lead connect to a resistivity monitor 112, as shown in Fig. 10B, which measures the resistivity across the sample, through a sliding brush 110a and ground wire. The resistivity readings from the monitor 112 are stored in the computer/controller 52 along with the saturation images.

The sample may be initially saturated with electrically conductive brine of known resistivity. During centrifuging the brine is displaced with nonconductive gas or oil and the sample resistivity increases. Alternatively, the centrifuging can be done in the imbibition mode with brine displacing nonconductive fluid. By combining the resistivity measurements with the saturation images in the computer/controller 52 the resistivity versus saturation curve is obtained for either drainage or imbibition cycles. An important advantage of this invention is that the saturation distribution down the core sample is known from the saturation images so that the resistivity curve may be corrected for the effects of non-uniform saturation caused by capillary end effects, as is known in the art.

Referring now to Fig. 11, there may be seen an alternative embodiment of the present invention. For this embodiment the sample holder 120 is modified to contain thereon both the electromagnetic radiation source 131 and detector 132. This embodiment is not limited to a particular rotational speed of the centrifuge. More particularly, the holder 120 has a radioactive source 131 and a solid state array detector 132 mounted on opposite sides of a holder 120. The source 131 may be for example, but not limited to, an Americium 241 line source. Alternate gamma-ray sources may also be employed in other embodiments of the present invention. Other embodiments may employ a radioactive sample as the source of electromagnetic radiation, or a radioactive source may be included in the fluids used in the sample. Electrical slip rings may be employed to carry the signals from the detector 132 to the computer/controller 52 for processing,

as described hereinbefore. The detector 132 may be a phosphor screen and photodiode array, as noted hereinbefore, or a solid state gamma-ray detector. In this embodiment, the radiation source and detector rotate with the sample to provide continuous data on fluid displacement and a strobed radiation system as described hereinbefore may not be needed, although it may also be used. By combining the continuous (mounted horizontally) and strobed (mounted vertically) features of the present invention, it is possible to construct images of the sample in three dimensions.

Referring now to Fig. 12, there may be seen an alternative embodiment based upon known principles. This embodiment is in essence the embodiment depicted in Fig. 1, but with the radiation source 24 now located below the sample holder 18 and the detector means 26 (screen 27, image intensifier 29, and camera 31) located immediately above sample holder 18, when sample holder 18 is in its "rotating" horizontal position. This embodiment also illustrates that the speed 54 and strobe 56 controller may be combined may be combined into one controller 56/54.

The apparatus of the present invention may be suitably employed to image a core sample from an earth formation undergoing various enhanced oil recovery techniques, such as, for example, but not limited to CO₂, steam and caustic recovery techniques. These samples can be exposed to high pressure and temperature representative of the reservoir conditions during such imaging, as noted hereinbefore. Such imaging provides valuable information on the actual fluid displacement processes occurring in the sample. Further, such images may be compared to images created by numerical simulations run in a high-speed computer, as described hereinbefore. This comparison may be through animated movies, video tape, or colour cathode ray tube displays. Further, the apparatus may be employed to determine variations in fluid flow properties throughout inhomogeneous samples. This eliminates any assumption of homogeneous fluid flow through a sample, and allows for the observation of any preferential channels or fractures in the sample.

Many other variations and modifications may be made in the apparatus and techniques hereinbefore described by those having experience in this technology without departing from the concept of the present invention.

Claims

1. A method for measuring the distribution of fluids in a porous sample, comprising the steps of: centrifuging said sample; periodically imaging said sample in a plurality of points during centrifuging; characterized by the additional step of measuring the resistivity of said sample during said centrifuging.
2. The method as claimed in claim 1, characterized by the step of calculating a resistivity versus saturation curve for said sample based upon said measured fluid distributions.
3. The method as claimed in claim 2, characterized by the step of correcting said resistivity versus saturation curve for capillary end effects based upon said imaging.
4. The method as claimed in any one of claims 1-3, characterized by the step of subjecting said sample to pressure during said centrifuging.
5. The method as claimed in claim 4, characterized in that said sample is subjected to overburden pressure.
6. The method as claimed in claim 4, characterized in that said sample is subjected to fluid pressure from an injected fluid.
7. The method as claimed in any one of claims 1-6, characterized by the step of visually displaying said distribution of fluids in said sample.
8. The method as claimed in any one of claims 2-7, characterized in that the step of calculating fluid saturations at said plurality of points.
9. The method as claimed in claim 8, characterized by the step of generating an animated movie of changes in fluid saturations.
10. The method as claimed in claim 9, characterized by the step of comparing said generated animated movie with a computer-generated numerical simulation animated movie of predicted changes in fluid saturations.
11. An apparatus for measuring the distribution of fluids in a sample, comprising a centrifuge (10) having a predetermined number of sample holders (18; 89) for containing said sample and capable of operating at plurality of speeds; a source (24) of electromagnetic energy positioned to radiate onto at least one preselected holder (18; 89); a detector array (26) positioned to detect electromagnetic energy transmitted through said at least one preselected holder (18; 89); means (22, 52, 54, 56, 58, 60) for periodically imaging of the said sample in a plurality of points during centrifuging; characterized in that the sample holders (89) are adapted to allow resistivity measurements during the centrifuging process.

as described hereinbefore. The detector 132 may be a phosphor screen and photodiode array, as noted hereinbefore, or a solid state gamma-ray detector. In this embodiment, the radiation source and detector rotate with the sample to provide continuous data on fluid displacement and a strobed radiation system as described hereinbefore may not be needed, although it may also be used. By combining the continuous (mounted horizontally) and strobed (mounted vertically) features of the present invention, it is possible to construct images of the sample in three dimensions.

Referring now to Fig. 12, there may be seen an alternative embodiment based upon known principles. This embodiment is in essence the embodiment depicted in Fig. 1, but with the radiation source 24 now located below the sample holder 18 and the detector means 26 (screen 27, image intensifier 29, and camera 31) located immediately above sample holder 18, when sample holder 18 is in its "rotating" horizontal position. This embodiment also illustrates that the speed 54 and strobe 56 controller may be combined may be combined into one controller 56/54.

The apparatus of the present invention may be suitably employed to image a core sample from an earth formation undergoing various enhanced oil recovery techniques, such as, for example, but not limited to CO₂, steam and caustic recovery techniques. These samples can be exposed to high pressure and temperature representative of the reservoir conditions during such imaging, as noted hereinbefore. Such imaging provides valuable information on the actual fluid displacement processes occurring in the sample. Further, such images may be compared to images created by numerical simulations run in a high-speed computer, as described hereinbefore. This comparison may be through animated movies, video tape, or colour cathode ray tube displays. Further, the apparatus may be employed to determine variations in fluid flow properties throughout inhomogeneous samples. This eliminates any assumption of homogeneous fluid flow through a sample, and allows for the observation of any preferential channels or fractures in the sample.

Many other variations and modifications may be made in the apparatus and techniques hereinbefore described by those having experience in this technology without departing from the concept of the present invention.

Claims

1. A method for measuring the distribution of fluids in a porous sample, comprising the steps of: centrifuging said sample; periodically imaging said sample in a plurality of points during centrifuging; characterized by the additional step of measuring the resistivity of said sample during said centrifuging.
2. The method as claimed in claim 1, characterized by the step of calculating a resistivity versus saturation curve for said sample based upon said measured fluid distributions.
3. The method as claimed in claim 2, characterized by the step of correcting said resistivity versus saturation curve for capillary end effects based upon said imaging.
4. The method as claimed in any one of claims 1-3, characterized by the step of subjecting said sample to pressure during said centrifuging.
5. The method as claimed in claim 4, characterized in that said sample is subjected to overburden pressure.
6. The method as claimed in claim 4, characterized in that said sample is subjected to fluid pressure from an injected fluid.
7. The method as claimed in any one of claims 1-6, characterized by the step of visually displaying said distribution of fluids in said sample.
8. The method as claimed in any one of claims 2-7, characterized in that the step of calculating fluid saturations at said plurality of points.
9. The method as claimed in claim 8, characterized by the step of generating an animated movie of changes in fluid saturations.
10. The method as claimed in claim 9, characterized by the step of comparing said generated animated movie with a computer-generated numerical simulation animated movie of predicted changes in fluid saturations.
11. An apparatus for measuring the distribution of fluids in a sample, comprising a centrifuge (10) having a predetermined number of sample holders (18; 89) for containing said sample and capable of operating at plurality of speeds; a source (24) of electromagnetic energy positioned to radiate onto at least one preselected holder (18; 89); a detector array (26) positioned to detect electromagnetic energy transmitted through said at least one preselected holder (18; 89); means (22, 52, 54, 56, 58, 60) for periodically imaging of the said sample in a plurality of points during centrifuging; characterized in that the sample holders (89) are adapted to allow resistivity measurements during the centrifuging process.



12. The apparatus as claimed in claim 11, characterized in that the sample holder (89) contains a plurality of electrodes (90) between which the resistivity is measured.

13. The apparatus as claimed in claim 12, characterized in that the said electrodes (90) consist of platinized platinum screen.

5

Patentansprüche

1. Verfahren zum Messen der Verteilung von Fluiden in einer porösen Probe, mit den Arbeitsschritten:

- Zentrifugieren der Probe,
- periodisches Abbilden der Probe an einer Vielzahl von Stellen während des Zentrifugierens, gekennzeichnet durch den zusätzlichen Arbeitsschritt
- Messen des spezifischen Widerstands der Probe während des Zentrifugierens.

2. Verfahren nach Anspruch 1, gekennzeichnet durch den Arbeitsschritt Berechnen einer Widerstand/Sättigungs-Kurve für die Probe, ausgehend von den gemessenen Fluidverteilungen.

3. Verfahren nach Anspruch 2, gekennzeichnet durch den Arbeitsschritt Korrigieren der Widerstand/Sättigungs-Kurve für kapillare Endeffekte, ausgehend von der Abbildung.

4. Verfahren nach einem der Ansprüche 1 bis 3, gekennzeichnet durch den Arbeitsschritt Beaufschlagen der Probe mit Druck während des Zentrifugierens.

5. Verfahren nach Anspruch 4, dadurch gekennzeichnet, daß die Probe Überlagerungsdruck ausgesetzt wird.

6. Verfahren nach Anspruch 4, dadurch gekennzeichnet, daß die Probe Fluidruck eines eingespritzten Fluids ausgesetzt wird.

7. Verfahren nach einem der Ansprüche 1 bis 6, gekennzeichnet durch den Arbeitsschritt optisches Anzeigen der Verteilung von Fluiden in der Probe.

8. Verfahren nach einem der Ansprüche 2 bis 7, gekennzeichnet durch den Arbeitsschritt Berechnen von Fluidsättigungswerten an der Vielzahl von Stellen.

9. Verfahren nach Anspruch 8, gekennzeichnet durch den Arbeitsschritt Erzeugen eines Zeichentrickfilms von Veränderungen der Fluidsättigungswerte.

10. Verfahren nach Anspruch 9, gekennzeichnet durch den Arbeitsschritt Vergleichen des erzeugten Zeichentrickfilms mit einem computererzeugten digitalen Simulations-Trickfilm von vorausgesagten Veränderungen der Fluidsättigungswerte.

11. Vorrichtung zum Messen der Verteilung von Fluiden in einer Probe, mit

- einer Zentrifuge (10), die eine vorbestimmte Anzahl von Probenaufnahmen (18; 89) zur Aufnahme der Probe aufweist und mit einer Vielzahl von Geschwindigkeiten betreibbar ist,
- einer Quelle (24) für elektromagnetische Energie, die so angeordnet ist, daß sie wenigstens eine im voraus bestimmte Aufnahme (18; 89) bestrahlt,

- einer Detektor-Gruppe (26), die so angeordnet ist, daß sie elektromagnetische Energie feststellt, die durch die wenigstens eine Aufnahme (18; 89) hindurchgeht,

- einer Einrichtung (22, 52, 54, 56, 58, 60) zum periodischen Abbilden der Probe an einer Vielzahl von Stellen während des Zentrifugierens,

dadurch gekennzeichnet, daß die Probenaufnahmen (89) Messungen des spezifischen Widerstands während des Zentrifugiervorganges ermöglichen.

12. Vorrichtung nach Anspruch 11, dadurch gekennzeichnet, daß die Probenaufnahmen (89) eine Vielzahl von Elektroden (90) enthalten, zwischen denen der spezifische Widerstand gemessen wird.

13. Vorrichtung nach Anspruch 12, dadurch gekennzeichnet, daß die Elektroden (90) aus platinisiertem Platin-Gitter bestehen.

Revendications

1. Une méthode pour mesurer la distribution de fluides dans un échantillon poreux, comportant les étapes de:

- centrifuger ledit échantillon;
- représenter de façon périodique ledit échantillon en une pluralité d'endroits pendant la centrifugation;

caractérisée par l'étape supplémentaire de:

- mesurer la résistivité dudit échantillon pendant ladite centrifugation.

2. La méthode telle que revendiquée dans la revendication 1, caractérisée par l'étape de calculer une courbe de la résistivité en fonction de la saturation pour ledit échantillon, en se basant sur lesdites distributions de fluides mesurées.

3. La méthode telle que revendiquée dans la revendication 2, caractérisée par l'étape de corriger ladite courbe de résistivité par rapport à la saturation pour des effets terminaux capillaires, en se basant sur ladite représentation d'image.

4. La méthode telle que revendiquée dans l'une quelconque des revendications 1 à 3, caractérisée par l'étape de soumettre ledit échantillon à une pression pendant ladite centrifugation.



5. La méthode telle que revendiquée dans la revendication 4, caractérisée en ce que ledit échantillon est soumis à une pression de surcharge.

6. La méthode telle que revendiquée dans la revendication 4, caractérisée en ce que ledit échantillon est soumis à une pression de fluide au moyen d'un fluide injecté.

5 7. La méthode telle que revendiquée dans l'une quelconque des revendications 1 à 6, caractérisée par l'étape d'afficher visuellement ladite distribution de fluide dans ledit échantillon.

8. La méthode telle que revendiquée dans l'une quelconque des revendications 2 à 7, caractérisée par l'étape de calculer les saturations en fluide de ladite pluralité d'endroits.

9. La méthode telle que revendiquée dans la revendication 8, caractérisée par l'étape de produire une représentation animée des modifications de saturation des fluides.

10 10. La méthode telle que revendiquée dans la revendication 9, caractérisée par l'étape de comparer ladite représentation animée produite avec une représentation animée par simulation numérique obtenue par l'ordinateur des modifications théoriques des saturations en fluide.

11. Un appareil pour mesurer la distribution des fluides dans un échantillon, comportant:

- 15 - une centrifugeuse (10) présentant un nombre prédéterminé de supports d'échantillon (18; 89) destiné à contenir ledit échantillon et susceptible de fonctionner à une pluralité de vitesses;
- une source (24) d'énergie électro-magnétique, positionnée pour irradier au moins un support pré-
- choisi (18; 89);
- 20 - un réseau détecteur (26) positionné pour détecter l'énergie électro-magnétique transmise à travers au moins un desdits supports préchoisis (18; 89);
- un dispositif (22, 52, 54, 56, 58, 60) pour représenter périodiquement l'image dudit échantillon en une pluralité d'endroits pendant la centrifugation;

caractérisé en ce que les supports d'échantillon (89) sont adaptés pour permettre des mesures de résistivité pendant la technique de centrifugation.

25 12. L'appareil tel que revendiqué dans la revendication 11, caractérisé en ce que le support d'échantillon (89) contient une pluralité d'électrodes (90) entre lesquelles la résistivité est mesurée.

13. L'appareil tel que revendiqué dans la revendication 12, caractérisé en ce que lesdites électrodes (90) sont constituées d'un écran platiné en platine.

