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Method and device for the control of surface properties of porous solids at the boundary of their contact with liquids and gases

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Abstract

The article indicates the relevance of the problem of controlling the surface properties of solids, analyzes the features of interaction between porous solids in direct contact with liquids.

The process of adhesive interaction of the system "liquid – porous solid" at the interface of these phases is analyzed and the dependence of the degree of wetting by a liquid of the surface of a solid on the structure of the porous body and the surface properties of the liquid is established. The dependence of the contact angle hysteresis of the solid with liquid on the porosity and roughness of the sample of the controlled body is substantiated.

A method of complex express control of the wetting process, which consists in determining the hysteresis of fluid flowing in and out from the surface of a solid body, has been proposed. The method consists in determining the rate of liquid outflow from a tilted sample of a solid. At the same time, the liquid is applied with the same speed to the surface of a porous body sample. The design of device for realization of the method has been developed.

Keywords: contact angle; porous solids; hysteresis; wetting; inflow; outflow.

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1. Introduction

The surface properties of solid materials determine the course of many natural and artificial technological processes. The interaction of solids with liquids and gases is determined primarily by the solids properties [1]. The study of these properties is necessary both for the possibility of controlling the oil recovery rocks processes, and for the creation of artificial biological tissues, varnishes, paints, lubricants, and others. In this case, the solid can be either a monolithic solid with a smooth surface (for example, metals after appropriate machining), or a porous body (for example, an oil and gas layer).

Especially important is the task of studying the surface properties of solid porous bodies, it depends not only on the chemical composition of the body substance, but also on its structure, the pores size. The solid surface tension is determined by the interaction of the solid surface with the gas or liquid surface, at the interface of these phases. Therefore, the study of the solid surfaces is carried out by wetting this surface by liquids.

Solids are characterized by irregularity (diffusion and chemical), the degree of which is usually unknown. Therefore, in each **specific** experiment it is a surface tension of the given sample, instead of substance in general. Even with perfect purity of the solid, its structure contains vacancies and dislocations ("impurities") that disrupt the normal structure of the crystal lattice. The solid surface roughness also has a significant effect on the surface tension value. In addition, it is for a solid body that the mechanical γ (which determines the elastic surface properties) and thermodynamic σ (which determines the energy properties) surface tensions do not coincide [2].

It is especially important to study the oil rocks surface properties. To increase their oil recovery, hydrophilization and oleophobization of the hard rock surface are required. The directions of these processes are determined by control the rock surface during its interaction with the liquid.

In the devices manufacture such as "artificial heart" and "artificial kidney", and in solving other practical problems of medicine (for example, blood transfusion) must take into account the physical-chemical phenomena that occur at the moment blood comes into contact with a solid surface.

Therefore, the purpose of this work is to develop a method for measuring the Contact Angle of solid porous. It is necessary to develop a method that would allow rapid measurements without complying with the complex requirements for sample purity, environmental conditions and to assess the wetting degree of the solid

Table 1

Contact angle hysteresis at different ratios f_P / f_S and protrusions heights h

h, mm	f_P / f_S			
	20	40	60	80
0.25	—	3	9	4
0.8	12	20	23	21
1.5	31	41	43	32
2.5	49	53	57	50

by a parameter that is convenient for further processing and interpretation.

2. Improved method and device

The porous body wetting occurs when the interaction energy of solid and liquid surfaces changes, thermodynamically inverted, and the free energy of the system is released in the form of work, which is completely spent on overcoming internal friction during fluid movement in the pores. For the theoretical substantiation of this process, two conditions are introduced: wetting is carried out at a finite speed; the pores are completely filled with liquid and after wetting they do not have air bubbles.

The contact angle of the porous surface can be determined by the following formula:

$$\cos\theta_P = f_S \cos\theta - f_P, \qquad (1)$$

where f_S, f_P – the area of the parts surface occupied by the solid material and pores, respectively; θ, θ_P – contact angles of the solid surface, occupied respectively by solid material and pores.

To confirm the formula (1) in [3] were conducted studies of water wetting of various polymeric surfaces made of polyethylene, polypropylene and Teflon, as well as wax. As a porous body, a model consisting of tubes set, cylinders and prisms arranged in a certain order with a fixed gap between the individual elements was proposed. For such a surface were calculated: free energy as a function of porosity, the contact angle on a smooth solid surface with a constant volume of the drop, as well as the actual drop contact angle on the porous surface and its hysteresis during inflow and outflow. The drop volume of water was 0.05 ml, the marginal contact angle on a solid surface was 95°. The studies were performed for different protrusions sizes on the solid surface samples (0.25–2.5 mm) and different ratios of areas f_P / f_S (20, 40, 60, 80). The hysteresis dependence of the contact angles on the solid porosity (f_P / f_S), is shown in Table 1.

Depending on the ratio f_P / f_S, the contact angle hysteresis changes. The maximum hysteresis is observed when the ratio f_P / f_S becomes equal to 60. The greater the height of the porous surface protrusions, the greater the hysteresis.

In wetting porous solids, it is necessary to take into account that the contact angle in these conditions differs significantly from the contact angle in wetting smooth surfaces. The approximate values of the contact angle obtained by wetting various porous bodies are as follows: for water – 67–84°; for kerosene – 65–71°; for carbon tetrachloride – 55–67° [4].

In directly measuring the contact angle of porous bodies, it is necessary to use drops commensurate with the pore size. This is not always possible on practice. In measuring the wetting of porous bodies, additional difficulties arise when uneven wetting of the surface elements of this body is detected. Therefore, porous bodies wetting is often determined by indirect methods [5].

Therefore, a method that combines the process of liquid inflow and outflow, i.e. contact angle hysteresis determination of the porous body surface is proposed. The liquid is applied dropwise to a sample of a porous body inclined at a certain angle. The inclination of the test sample leads to the outflow of liquid from its surface with partial penetration into its pores. When studying solids of different structure and chemical

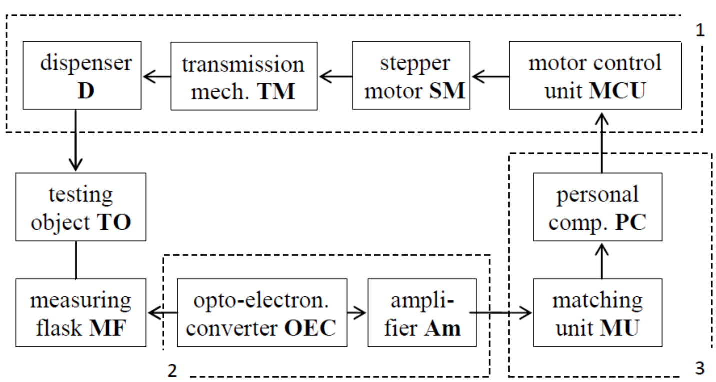

Fig. 1. Block diagram of the developed device

composition, the dimensions of their samples and the liquids volumes applied to them must be the same.

According to this method, the liquid wetting process of a porous solid sample is evaluated based on the change speed determination of the liquid volume, that flowing out of the inclined sample. This method allows you to evaluate pure liquids and solutions of different chemical composition.

The outflow speed is monitored by continuously recording the change in the test fluid volume that flows from the inclined sample surface and enters an optically transparent measuring container. The block diagram of the device that implements the proposed method is shown in Fig. 1.

Structurally, the device is made in the form of three separate, cable-connected modules:

1 – node of precise movement of the batcher piston (electromechanical block);

2 – node of measurement of filling volume by the investigated liquid of measuring capacity (measuring block);

3 – node of volume change registration in time and comparison of the received curves of outflow liquid dynamics and control of the batcher work (the processing and control block).

Node 1 includes a four-winding stepper motor SM, it through the transmission mechanism TM discretely moves up and down the piston of the dispenser D to apply the test liquid to the sample surface, that is the testing object TO. The sample is mounted on a base (table) inclined at an angle of 15°, to the lower end is attached a glass measuring flask MF. The operation of the motor is controlled by a Personal Computer PC using the motor control unit MCU alternately supply to its windings pulses of voltage 5 V, current 1 A and a length (pulse duration) of about 10 ms. The stepper motor used in the device applying one pulse to the corresponding winding makes it possible to obtain a minimum angle of rotation on its shaft equal to 108 angular minutes.

The MCU unit on the PC command switches the motor windings to turn it on, reverse or stop, as well as prevents the passage of pulse interference to other device units due to the operation of high current motor windings. TM is designed to transmit the rotational motion of the motor shaft in the piston translational motion (when the shaft rotates 360°, the metering piston moves by 0.5 mm).

The design of node 1 with the testing object and the measuring container is presented in Fig. 2.

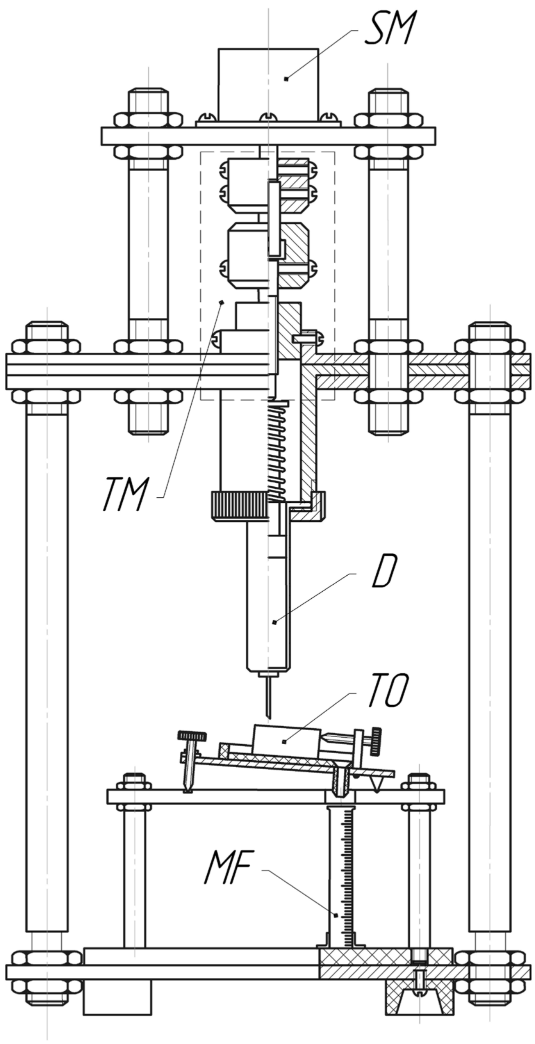

Fig. 2. Construction of an electromechanical block with the testing object and the measuring container

Node 2 consists of an opto-electronic converter OEC, the signal from it fed to the amplifier Am and through the matching unit MU is registered in the PC.

Node 3 contains a PC with a matching unit MU. The MU unit is designed to provide two-way communication between the PC and other device units. The sample number and the liquid type, as well as all the necessary commands about the beginning and end of the measurement are entered via the keyboard. The PC processes the information about the liquid volume change in time in the MF, compares the obtained time dependences with stored in its memory and draws a conclusion about the wetting degree of the porous solid, as well as controls the device operation.

The developed device allows express control the wetting degree of the porous body by the test liquid determining the change speed of the liquid volume that flowing from the inclined test sample after its drop application to the sample surface. The higher the flow liquid speed into the measuring flask, i.e. the greater the inclination curve angle of the liquid amount change in time in the measuring contain, the smaller contact angle, which corresponds to the higher wetting degree. The graphical dependence of the liquid volume change in time obtained in the PC is compared with the theoretical dependences, the stored in the PC memory, to select the closest one, which corresponds to a certain value of the contact angle.

3. Conclusion

Based on the process analysis of liquid inflow and outflow from the porous solid surface is proposed to carry out express control of the process liquid wetting of the porous solid surface by contact angle hysteresis, i.e. the flow speed of the test fluid. The liquid is dosed applied with a constant speed on the investigated inclined sample surface.

The design of the device is implemented, which implements the proposed method and allows to study the influence of the parameters of the system "solid – liquid" on the wetting process and to make the optimal choice of pure liquid or solution for wetting of the specific porous solid body.

Метод та пристрій контролю поверхневих властивостей твердих пористих тіл на межі їх контакту з рідинами і газами

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Анотація

У статті вказано актуальність проблеми контролю поверхневих властивостей твердих тіл, визначено особливості взаємодії пористих твердих тіл при їх безпосередньому контакті з рідинами і газами. Проаналізовано процес адгезійної взаємодії системи "рідина – пористе тверде тіло" на межі розділу цих фаз і встановлено однозначну залежність ступеня змочування рідиною поверхні твердого тіла від структури пористого тіла, його шорсткості та поверхневих властивостей рідини і газу. Проаналізовано значення гістерезису кута змочування, тобто різниці між кутами натікання та відтікання чистих рідин і розчинів поверхнево-активних речовин із поверхонь твердих тіл різної структури. Обґрунтовано залежність гістерезису кута змочування твердого тіла рідиною від пористості та шорсткості зразка контрольованого тіла. Запропоновано метод комплексного експрес-контролю процесу змочування, який полягає у визначенні гістерезису натікання і відтікання рідини з поверхні твердого тіла. Метод передбачає визначення швидкості зміни об'єму рідини, що відтікає із поверхні нахиленого зразка досліджуваного твердого тіла та після проходження через його пори. При цьому рідина наноситься з однаковою швидкістю на поверхню зразка пористого тіла, розміри зразків досліджуваних твердих пористих тіл та їх кут нахилу є однаковими для всіх досліджень. Розроблено конструкцію пристрою для реалізації методу, в якому передбачено прецизійне переміщення поршня дозатора для нанесення рідини на досліджувану поверхню, неперервне вимірювання і реєстрація об'єму наповнення мірної ємності рідиною, що натекла із поверхні зразка або пройшла через пори, та порівняння кривих динаміки відтікання рідини для встановлення значення крайового кута змочування і оптимального вибору типу чистої рідини або розчину для змочування конкретного пористого тіла.

Ключові слова: крайовий кут змочування; пористі тверді тіла; гістерезис; змочування; натікання; відтікання.

Метод и устройство контроля поверхностных свойств твердых пористых тел на границе их контакта с жидкостями и газами

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Аннотация

В статье указана актуальность проблемы контроля поверхностных свойств твердых тел, проанализированы особенности взаимодействия пористых твердых тел при непосредственном контакте с жидкостями.

Проанализирован процесс адгезионного взаимодействия системы “жидкость – пористое твердое тело” на границе раздела этих фаз и установлена зависимость степени смачивания жидкостью поверхности твердого тела от структуры пористого тела и поверхностных свойств жидкости. Обоснована зависимость гистерезиса угла смачивания твердого тела жидкостью от пористости и шероховатости образца контролируемого тела.

Предложен метод комплексного экспресс-контроля процесса смачивания, который заключается в определении гистерезиса натекания и оттекания жидкости с поверхности твердого тела. Метод заключается в определении скорости оттекания жидкости из наклоненного образца твердого тела. При этом жидкость наносится с одинаковой скоростью на поверхность образца пористого тела. Разработана конструкция устройства для реализации метода.

Ключевые слова: краевой угол смачивания; пористые твердые тела; гистерезис; смачивание; натекание; оттекание.

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