INNOVATIVE TECHNOLOGIES FOR OIL AND GAS RESOURCES EXPLOITATION
IN COMPLICATED GEOLOGICAL AND CRITICAL ENVIRONMENTAL CONDITIONS

Original article

Study of the properties of air nanobubbles in water obtained using the Anopore membrane

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Abstract. *Background*. The paper presents an original method for generating air nanobubbles in water. Research into nanobubble generation methods is of current interest due to their unique properties and potential applications in various fields, including oil and gas. Traditional methods for nanobubble production are often complex, which stimulates the search for new, simpler approaches. *Objective*. To describe and experimentally validate a new, simple method for generating air nanobubbles in water by filtration through a dry nanoporous membrane. *Materials and methods*. For nanobubble generation, deionized water was filtered through a dry syringe filter with an Anopore membrane (pore diameter 20 nm). Detection and characterization of nanobubbles were performed using dynamic light scattering and ultramicroscopy. Nanoparticle tracking analysis was used for independent determination of nanobubble sizes. *Results*. It was shown that the proposed method leads to nanobubble formation. The average nanobubble radius immediately after filtration was about 50 nm, increasing to 130–150 nm over 30 min. The concentration of nanobubbles increased by an order of magnitude within 20 min. Nanobubble formation was not observed when using a prewetted Anopore filter or filters made of polyethersulfone. *Conclusions*. A simple and reproducible method for generating nanobubbles based on water filtration through a dry Anopore membrane was developed. The presence and characteristics of nanobubbles were confirmed by independent experimental methods. Further research is required for a complete understanding of the nanobubble formation mechanism and their properties.

Keywords: nanobubbles, ultramicroscopy, Anopore membrane, dynamic light scattering

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Introduction

Nanobubbles (NBs) are gas-filled cavities with diameters typically below 200 nm, exhibiting unique physicochemical properties due to their nanoscale dimensions. Unlike conventional microbubbles, NBs demonstrate exceptional stability in liquid media, persisting for weeks or even months. The extremely large surface area-to-volume ratio of NBs enhances gas transfer efficiency, making them valuable in biomedical, environmental and industrial applications. Their behavior defies classical bubble dynamics, as they exhibit anomalous mobility and interfacial interactions that remain subjects of active research.

Nanobubbles can be generated through various methods, including ultrasonic cavitation, membrane diffusion and electrochemical processes [1]. Their applications span multiple fields: biomedicine (contrast-enhanced imaging, drug delivery, tissue oxygenation) [2, 3]; water treatment (pollutant degradation, disinfection) [4]; agriculture (enhanced plant growth, precision agrochemical delivery) [5]; mining and industry (improved froth flotation, surface cleaning, water preparation and others) [6]. The field of nanobubble research is relatively young. Due to the difficulties in obtaining and detecting NBs, there are few articles on this topic, but their number is growing every year.

Some of the first theoretical descriptions of NBs were made in the work [7]. Epstein and Plesset formulated a pioneering theoretical model describing gas bubble dissolution in liquids, combining diffusion theory with the Laplace pressure equation. According to the Epstein–Plesset theory, a bubble with a radius of 100 nm will have an internal pressure approximately 14.4 times higher than atmospheric, and its lifetime cannot exceed 1 ms, which contradicts experiments in which the NB lifetime is significantly longer, up to several days. The paradox between the short lifetime predicted by the Epstein–Plesset theory and the experimentally observed long lifetime of NBs in water has not yet been resolved [8–10]. There are several theories explaining the possibility of the existence of long-lived NBs [11, 12].

Of particular interest is the potential use of nanobubbles in the oil and gas industry, where efficient flow control and enhanced oil recovery are critically important. The following are the main promising areas of application for nanobubbles in the oil and gas industry. Enhanced oil recovery: nanobubbles can reduce oil viscosity by dissolving gas, create “gas traffic” to improve mobility and change rock wettability to be more hydrophilic, facilitating the detachment of oil droplets from the rock surface [13]. There are studies that show that CO_2 NBs can enhance oil recovery in tight shale reservoirs [14]. However, the implementation of nanobubble technologies in oil and gas practice is associated with a number of fundamental and technological challenges. Key among these is the issue of nanobubble stability under reservoir conditions (high temperatures, pressures, formation water salinity), which can significantly limit their lifetime and effectiveness [15]. Furthermore, questions related to the behavior of nanobubbles in porous media remain open: the mechanisms of their transport, retention and interaction with fluids and rock are insufficiently studied. The problem of large-scale, cost-effective and stable generation of nanobubbles directly at the field level has not been fully resolved. Thus, despite their significant potential, the use of nanobubbles in the oil and gas industry requires in-depth research aimed at understanding their behavior under real reservoir conditions, developing reliable generation methods and optimizing their application technologies to address specific production challenges.

The objective of the paper is to describe and experimentally validate a new method for producing air nanobubbles in water. This method is significantly simpler than other methods.

Material and methods

To detect NBs, we used the dynamic light scattering (DLS) method (via Photocor Compact-Z, Russia), which allows measuring the size of nanoobjects in liquid, and the ultramicroscopy method (via NP Counter, Russia)[1], which, due to special illumination of the sample with a laser (analogous to the dark-field method in optical microscopy), allows visualizing nanoobjects in liquid (from 10 nm), observing their movement and measuring their numerical concentration.

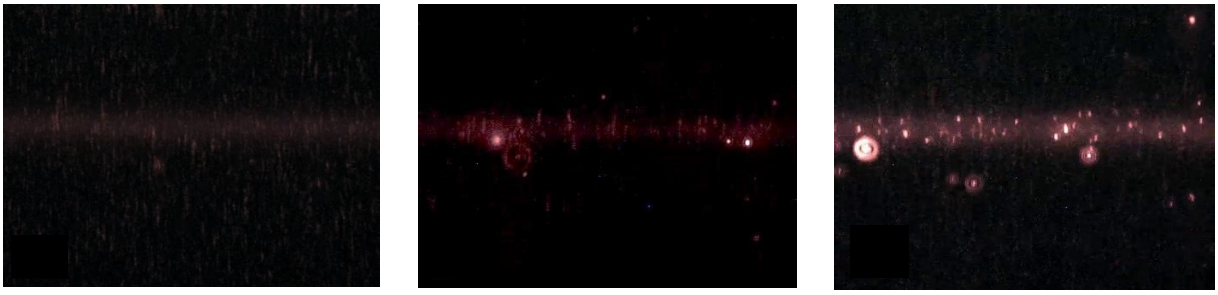
The proposed new method for obtaining air nanobubbles in water consists in filtering a small volume (2–3 ml) of distilled, deionized, purified by reverse osmosis water (Solopharm medical water for injection, Russia) through a dry syringe filter with a pore diameter of 20 nm. To obtain NBs, a Whatman Anotop 10 syringe filter containing an inorganic Anopore membrane was used. During filtering, it was necessary to apply force to the syringe plunger in order to force the water through the filter. Immediately after filtration, a portion of the water sample (approximately 1 ml) was placed in a cuvette in the DLS device, and another portion (approximately 1 ml) in a separate cuvette in the ultramicroscopy device. Every minute, measurements were taken of the scattered light intensity, the correlation function of the scattered light intensity fluctuations (hydrodynamic radius of the particles) and the numerical concentration of the particles.

Results and discussion

The initial water sample was preliminarily examined by DLS and ultramicroscopy. The scattering intensity on the water sample before filtration was 5,100±100 cps (counts per second), and correlation functions indicating the presence of particles in the water were not observed. During the examination of the initial water by ultramicroscopy, particles (background contamination) periodically entered the field of view of the device; the concentration of such particles was estimated at about 10^6 pcs/ml.

Fig. 1 shows images of a typical ultramicroscope view when observing clean water (a), a water sample immediately after filtration through a 20 nm filter (b) and a water sample 10 min after filtration (c). In each of the images, a light horizontal line can be seen in the center – this is the scattering of the focused laser beam on water molecules (Rayleigh scattering). Only in this area, in a small part of the entire sample volume illuminated by the laser, objects are observed and counted. This volume is 1.2×10^{-7} ml. In Figs. 1b and 1c, there are bright light dots in the images – this is scattering on individual nanosized objects in water. We assume that these are air nanobubbles formed as a result of filtering water through 20 nm filter pores. The filter was prewashed with water several times and the observed objects cannot be particles washed off the filter. It can be noted that immediately after filtration there are fewer such objects in the water than after 10 min. This can most likely be explained by the fact that the concentration and size of NBs increases with time after filtration. At the beginning of the experiment, the size and concentration of NBs are such that the ultramicroscopy and DLS methods register particles at the sensitivity limit of the devices used.

[1] NP Counter. URL: https://npcounter.ru/ (accessed 1 September 2025).

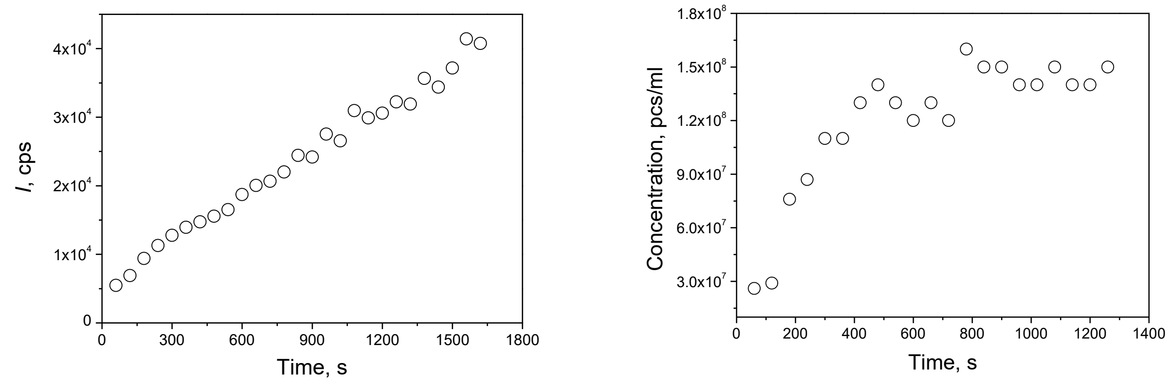


а/a б/b в/c

Fig. 1. Typical ultramicroscopic view of pure water (a),
a water sample immediately after filtration through a 20 nm Anopore membrane filter (b)
and a water sample 10 min after filtration (c)

Рис. 1. Типичный вид поля зрения при наблюдении в ультрамикроскоп чистой воды (а),
образца воды сразу после фильтрации через фильтр с мембраной Anopore 20 нм (б)
и образца воды через 10 мин после фильтрации (в)

For a water sample filtered through a 20 nm Anopore membrane filter, the scattered light intensity and numerical concentration of NBs were measured as a function of time. In 30 min, the scattered light intensity increased from 5,000 cps to 40,000 cps (Fig. 2a). The numerical concentration of NBs increased by almost an order of magnitude in 20 min and reached saturation (Fig. 2b).



а/a б/b

Fig. 2. Time dependence of the intensity of scattered light (a) and the concentration of NBs (b)
in a water sample after filtration

Рис. 2. Зависимость от времени интенсивности рассеянного света (а) и концентрации НП (б)
в образце воды после фильтрации

The increase in scattered light intensity may be due to both the growth in NBs size and concentration in the volume and the growth of macrobubbles on the walls of the cuvette, the appearance of which may lead to glare and affect the measured value of scattered light intensity. A photo of a cuvette with such surface macrobubbles on the walls of the cuvette is shown in Fig. 3. At the same time, the effect of such glare on the process of measuring the particle size by the DLS method (correlation functions of scattered light) is insignificant.

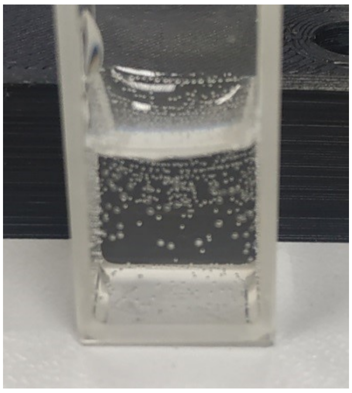


Fig. 3. Image of surface microbubbles on the cuvette walls

Рис. 3. Изображение поверхностных микропузырьков на стенках кюветы

Measurements of the sizes (hydrodynamic radius) of NBs by the DLS method show that immediately after filtration, the average radius of NBs is about 50 nm, and after 30 min, the average radius becomes 130–150 nm. Such sizes are typical for NB samples [1]. In this sample, the nanoparticle size was measured using nanoparticle tracking analysis (NTA). Fig. 4 shows the nanoparticle size distribution. The maximum of this distribution is about 150 nm, which is in good agreement with the average particle size in this sample obtained by dynamic light scattering (DLS).

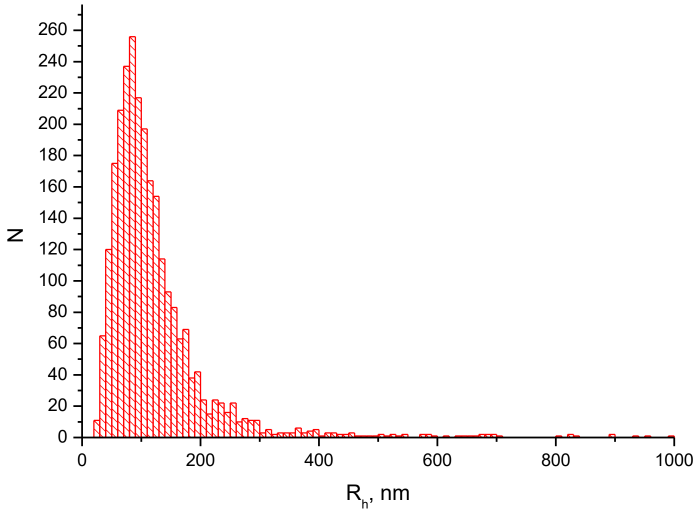


Fig. 4. Nanoparticle size distribution measured by nanoparticle tracking analysis

Рис. 4. Распределение наночастиц по размерам, измеренное методом анализа траекторий наночастиц

A day after sample preparation, the presence of NBs is not detected by the DLS method probably because of their low concentration. Using the ultramicroscopy method, objects can be detected in such a sample a day after preparation. Their concentration is about 10^7 pcs/ml, which is higher than the background values for the source water, but lower than the concentration of NBs in the first half hour after sample preparation.

It is important to note that after filtering water through a filter with 20 nm pores (Anopore membrane), after some time, macrobubbles visible to the naked eye appear on the walls of the cuvette. The filtration process leads to the appearance of air bubbles on the walls of the cuvette. Macrobubbles on the walls of the cuvette may be the result of the formation of surface nanobubbles [16] on the walls and their subsequent growth to macro sizes.

When filtering water through a prewetted filter, the formation of NBs was not observed by the ultramicroscopy and DLS methods. On the same filter, after drying, the formation of NBs was reproduced in more than three experiments and on two different filters. For 20 nm syringe filters with polyethersulfone (PES) as a filter element, the formation of neither nanobubbles in the volume by the ultramicroscopy method nor macrobubbles with the naked eye on the walls of the cuvette after filtration was observed. In such filters, the filter material differs significantly from the filter material (pore shape) of the Anopore membrane used in Whatman Anotop filters which has sharply defined capillary pore structure.

Apparently, when filtering water through a dry filter, when the filter pores contain some air, NBs are formed. Their size and concentration are such that immediately after filtration, their detection by the DLS and ultramicroscopy methods occurs at the limit of sensitivity of the devices used in the study and, accordingly, these parameters are measured with low accuracy. However, after a few minutes the NBs size increases, which leads to an increase in the scattering of laser radiation on NBs and the accuracy and reliability of measurements by the ultramicroscopy and DLS methods increases.

It is important to note that not all samples in which NBs were detected by the ultramicroscopy method after filtration could measure the NBs size by the DLS method. Apparently, it was not always possible to obtain a NBs concentration sufficient for measurements by the DLS method. An additional study was conducted to determine the sensitivity threshold of the DLS device used. Samples of aqueous solutions of SiO_2 nanoparticles (100 nm and 200 nm) and a colloidal gold sample (34 nm), in which the DLS method measures particle sizes (correlation functions of scattered light intensity fluctuations) with good accuracy and reproducibility, were titrated with pure water to a concentration of nanoparticles at which the DLS device does not allow measuring the particle size in these samples due to the low concentration. Then, the numerical concentration of nanoparticles was measured in such samples by ultramicroscopic analysis. For all samples, this concentration was about 10^8 pcs/ml. Thus, it can be stated that at concentrations of nanoobjects in water below this value, the DLS device used will not allow measuring the particle size. This was observed for some samples during experiments with NBs.

Conclusion

This paper describes a simple method for producing air nanobubbles in water. In studies published to date, generating nanobubbles using membranes with varying pore sizes involves forcing gas through the membranes into the aqueous phase. In the method proposed in this paper for producing nanobubbles, water is forced through the pores.

The presence of NBs in the samples was confirmed by two experimental methods (DLS and ultramicroscopy). The data obtained on the size and concentration of NBs in the investigated samples are in good agreement with the published data of other authors.

The described method for obtaining NBs is quite simple and easily reproducible, but for a better understanding of the mechanisms of NBs formation during water filtration through a 20 nm Anopore membrane filter and the study of the physicochemical properties of such NBs, additional research is required.

Author contributions

Vladimir N. Kuryakov – conceptualization, data curation, formal analysis, methodology, investigation, validation, visualization, writing original draft, writing – review & editing.

Conflict of interests

The author declares no conflict of interests.

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ИННОВАЦИОННЫЕ ТЕХНОЛОГИИ ОСВОЕНИЯ НЕФТЕГАЗОВЫХ РЕСУРСОВ В СЛОЖНЫХ ГОРНО-ГЕОЛОГИЧЕСКИХ И ЭКСТРЕМАЛЬНЫХ ПРИРОДНО-КЛИМАТИЧЕСКИХ УСЛОВИЯХ

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УДК 544.77
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Исследование свойств нанопузырьков воздуха в воде, полученных при помощи мембраны Anopore

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Аннотация. *Актуальность*. В работе представлено описание оригинального метода получения нанопузырьков воздуха в воде. Исследование методов генерации нанопузырьков актуально в связи с их уникальными свойствами и перспективами применения в различных отраслях, включая нефтегазовую. Традиционные способы получения нанопузырьков часто сложны, что стимулирует поиск новых, более простых подходов. *Цель работы*. Описание и экспериментальная проверка нового простого метода получения нанопузырьков воздуха в воде путем фильтрации через сухую мембрану с нанопорами. *Материалы и методы*. Для генерации нанопузырьков использовалась фильтрация деионизированной воды через сухой шприцевой фильтр с мембраной Anopore (диаметр пор 20 нм). Детектирование и характеристика нанопузырьков проводились методами динамического светорассеяния и ультрамикроскопии. Анализ траекторий наночастиц применялся для независимого определения размеров нанопузырьков. *Результаты*. Показано, что предложенный метод приводит к образованию нанопузырьков. Их средний радиус непосредственно после фильтрации составлял около 50 нм, увеличиваясь до 130–150 нм в течение 30 мин. Концентрация нанопузырьков возрастала на порядок за 20 мин. Образование нанопузырьков не наблюдалось при использовании предварительно смоченного фильтра или фильтров из полиэфирсульфона. *Выводы*. Разработан простой и воспроизводимый метод получения нанопузырьков, основанный на фильтрации воды через сухую мембрану Anopore. Наличие и характеристики нанопузырьков подтверждены независимыми экспериментальными методами. Для полного понимания механизма формирования нанопузырьков и их свойств требуются дальнейшие исследования.

Ключевые слова: нанопузырьки, ультрамикроскопия, мембрана Anopore, динамическое светорассеяние

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В.Н. Курьяков – концептуализация, администрирование данных, формальный анализ, методология, проведение исследования, верификация данных, визуализация, создание черновика рукописи, создание рукописи и ее редактирование.

Конфликт интересов

Автор заявляет об отсутствии конфликта интересов.

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