ISSN 2518-170X (Online)
ISSN 2224-5278 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫ
Satbayev University

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ
НАУК РЕСПУБЛИКИ
КАЗАХСТАН
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NEWS

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF
KAZAKHSTAN
Satbayev University

SERIES
OF GEOLOGY AND TECHNICAL SCIENCES

1 (457)

JANUARY – FEBRUARY 2023

THE JOURNAL WAS FOUNDED IN 1940

PUBLISHED 6 TIMES A YEAR

ALMATY, NAS RK

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NAS RK is pleased to announce that News of NAS RK. Series of geology and technical sciences scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of geology and technical sciences in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of geology and engineering sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы «ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы» ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді геология және техникалық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия геологии и технических наук» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК. Серия геологии и технических наук в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по геологии и техническим наукам для нашего сообщества.

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«ҚР ҰҒА Хабарлары. Геология және техникалық ғылымдар сериясы».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Меншіктеуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» РҚБ (Алматы қ.).
Қазақстан Республикасының Ақпарат және қоғамдық даму министрлігінің Ақпарат комитетінде 29.07.2020 ж. берілген **№ KZ39VPY00025420** мерзімдік басылым тіркеуіне қойылу туралы куәлік.
Тақырыптық бағыты: *геология, мұнай және газды өңдеудің химиялық технологиялары, мұнай химиясы, металдарды алу және олардың қосындыларының технологиясы.*
Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.
Редакцияның мекен-жайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., тел.: 272-13-19
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«Известия НАН РК. Серия геологии и технических наук».
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы).
Свидетельство о постановке на учет периодического печатного издания в Комитете информации Министерства информации и общественного развития Республики Казахстан **№ KZ39VPY00025420,** выданное 29.07.2020 г.
Тематическая направленность: *геология, химические технологии переработки нефти и газа, нефтехимия, технологии извлечения металлов и их соеденений.*
Периодичность: 6 раз в год.
Тираж: 300 экземпляров.
Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, оф. 219, тел.: 272-13-19
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Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75.

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News of the National Academy of Sciences of the Republic of Kazakhstan. Series of geology and technology sciences.
ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)
Owner: RPA «National Academy of Sciences of the Republic of Kazakhstan» (Almaty).
The certificate of registration of a periodical printed publication in the Committee of information of the Ministry of Information and Social Development of the Republic of Kazakhstan **No. KZ39VPY00025420,** issued 29.07.2020.
Thematic scope: *geology, chemical technologies for oil and gas processing, petrochemistry, technologies for extracting metals and their connections.*
Periodicity: 6 times a year.
Circulation: 300 copies.
Editorial address: 28, Shevchenko str., of. 219, Almaty, 050010, tel. 272-13-19
http://www.geolog-technical.kz/index.php/en/

© National Academy of Sciences of the Republic of Kazakhstan, 2023

Address of printing house: ST «Aruna», 75, Muratbayev str, Almaty.

NEWS of the National Academy of Sciences of the Republic of Kazakhstan
SERIES OF GEOLOGY AND TECHNICAL SCIENCES
ISSN 2224-5278
Volume 1, Number 457 (2023), 20-32
https://doi.org/10.32014/2023.2518-170X.256

UDC 532.536

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STUDY OF CARBONATE ROCK DISSOLUTION USING X-RAY MICROCOMPUTED TOMOGRAPHY: IMPACT OF ACID FLOW RATE

Abstract. High-pressure high-temperature flow apparatus in combination with X-ray micro-tomography were used to study the change of pore structure during the dissolution of carbonate core samples. 8 carbonate samples (with >99% calcite) were considered. During the dissolution experiment, an HCl solution with a concentration of 12 or 18% was injected into these core samples at flow rates of 1, 2, 4 and 8 ml/min. Samples were scanned before and after the acid injection at about 19 microns. Avizo® software was used for image processing.

Results show a significant increase in permeability due to dissolution, especially at high flow rates. It was shown that images with a resolution of about 19 μm are not enough to estimate overall porosity of the samples. Also, it has been shown that at this resolution the median filter is more suitable.

The change of the 3D pore structure due to dissolution shows that the dissolution pattern was conical for low flow rates (1 and 2 ml/min), but for high (4 and 8 ml/min) flow rates it becomes more dominant for both 12% and 18% HCl concentration. It has been observed that the flow rate significantly impacts the sample-averaged dissolution rate. With an increase in the flow rate, the sample-averaged dissolution rate increases.

Key words: reactive dissolution, X-Ray micro-computed tomography, imaging, image porosity.

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STUDY OF CARBONATE ROCK DISSOLUTION USING X-RAY MICROCOMPUTED TOMOGRAPHY: IMPACT OF ACID FLOW RATE

Аннотация. Бұл мақалада карбонатты үлгілердің кеуекті құрылымдарының өзгеруі олардың жоғары қысым мен температурада жұмыс істейтін қондырғыда еруі рентгендік микротомографияны қоса пайдалану арқылы зерттелді. Құрамында 99%-дан жоғары кальциты бар 8 карбонатты керн үлгілері қарастырылды. Эксперимент барысында осы үлгілерге 12 және 18% тұз қышқылының ерітінділері 1, 2, 4 және 8 мл/мин жылдамдықта айдалды. Сонымен қатар, үлгілер ерітінділерді айдағанға дейін және айдағаннан кейін кеңістіктік дәлдігі 19 микрон болатын рентгендік микротомографпен сканерленді. Сканерлеу нәтижесінде алынған суреттер Avizo® арнайы бағдарламалық пакетін пайдалана отырып өңделді.

Нәтижелерге сәйкес, абсолютті өткізгіштіктің тау жынысының еруі кезінде, әсіресе, жоғары жылдамдықтарда айтарлықтай артатындығын көрсетті. Рентгендік микротомографтың дәлдігі (19 мкм) зерттеуде қарастырылған карбонатты үлгілердің жалпы кеуектілігін анықтауға жеткіліксіз екендігі көрсетілді. Сонымен қатар, осындай дәлдікте ауытқуларды азайтуда медианды сүзгінің айтарлықтай қолайлы екендігі көрсетілді.

Қарастырылған үлгілердің ерігенне кейінгі 3D кеуекті құрылымын талдау еру пішінінң аз жылдамдықтарда (1 және 2 мл/мин) коникалық, ал үлкен жылдамдықтарда (4 және 8 мл/мин) тұз қышқылының 12% және 18% концентрациялы ерітінділері үшін оның едәуір дамыған түрге ие болатындығын көрсетті. Бұған қоса, қышқылды ерітіндіні айдау жылдамдығының үлгі бойынша орталанған тау жынысының еру жылдамдығына айтарлықтай әсер ететіндігі байқалды. Айдау жылдамдығы артқан сайын үлгі бойынша орталанған тау жынысының еру жылдамдығы артты.

Түйінді сөздер: еріту, рентгендік микрокомпьютерлік томография, визуализация, кескіннің кеуектілігі.

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ИССЛЕДОВАНИЕ РАСТВОРЕНИЯ КАРБОНАТНЫХ ПОРОД С ИСПОЛЬЗОВАНИЕМ РЕНТГЕНОВСКОЙ МИКРОКОМПЬЮТЕРНОЙ ТОМОГРАФИИ: ВЛИЯНИЕ СКОРОСТИ ПОТОКА КИСЛОТЫ

Аннотация. В настоящей статье раскрыто изменение поровой структуры карбонатных образцов при их растворении на установке высокого давления и температуры совместно с рентгеновской микротомографией. Были рассмотрены 8 карбонатных образцов с содержанием кальцита >99%. В ходе экспериментов в эти образцы были закачаны кислотные растворы с концентрацией соляной кислоты 12 и 18% при расходах 1, 2, 4 и 8 мл/мин. Также образцы были отсканированы до и после закачки растворов с помощью рентгеновского микротомографа с пространственным разрешением около 19 микронов. Полученные в результате сканирования изображения были обработаны с помощью программного обеспечения Avizo®.

Результаты показали значительное увеличение проницаемости из-за растворения породы, особенно при высоких скоростях закачки. Показано, что пространственное разрешение рентгеновского микротомографа в 19 мкм недостаточно для оценки общей пористости образцов. Также было показано, что при таком разрешении медианный фильтр являлся наиболее подходящим для уменьшения шумов.

Анализы 3D-поровой структуры рассмотренных образцов после растворения показали, что форма растворения была конической при низких скоростях закачки (1 и 2 мл/мин), но при высоких скоростях закачки (4 и 8 мл/мин) она становится более развитой (доминантные червоточины) как для 12% и 18% концентрации кислоты. Также было замечено, что скорость закачки значительно влияет на осредненную по образцу скорость растворения породы. С увеличением скорости закачки осредненная по образцу скорость растворения увеличивалась.

Ключевые слова: растворение, рентгеновская микрокомпьютерная томография, визуализация, пористость по изображению.

Introduction. The microstructure of carbonate rocks experiences substantial changes under a reactive processes, in particular chemical dissolution and deposition (Qajar et al., 2013). The concept behind the matrix acidizing is to create high conductive channels (wormholes) when hydrochloric (HCl) acid dissolves calcite layers in carbonate-rich formations. These wormholes will connect the near-wellbore zone to the reservoir through the damaged layer, and as a result, increase hydrocarbon production

(Turegeldieva et al., 2016). The acid type and its concentration must be carefully selected to achieve a successful acid treatment.

Rötting et al. investigated the changes in the initial permeability, porosity and pore size distribution during the dissolution of sedimentary carbonate rocks with dilute acid (HCl) (Rötting et al., 2015). Soltanbekova et al. concluded that the dissolution efficiency largely depends on the flow rate, rock permeability, rock lithology and other factors during the injection of 15% HCl into 6 different carbonate core samples (Soltanbekova et al., 2021).

It has been shown that the permeability of high porosity cores did not increase significantly during the long-term dissolution experiments (~1000 h), may be because the dissolution occurred primarily in the pores rather than in pore throats (Colón et al., 2004).

Molins et al. (2014) investigated the impact of transport and reaction on calcite dissolution at a pore-scale. Authors used a high-resolution pore-scale numerical model to simulate the experiment on a computational domain consisting of reactive calcite, pore space, and the capillary wall constructed from volumetric X-Ray micro-computed tomography (μ-CT) images (Molins et al., 2014). Noiriel et al. studied the dissolution of a limestone core during the CO_2-enriched water injection experiment (Noiriel et al., 2004). They measured the changes in porosity and permeability arising from modifications of the pore network geometry and the fluid-rock interface using the μ-CT imaging.

Jiang & Tsuji studied the influence of precipitation on relative permeability during CO_2 storage using numerical methods (Jiang & Tsuji, 2014). They first extracted the pore structure of Berea sandstone rock samples by high-resolution μ-CT scanned images, then calculated the relative permeability with a highly optimized two-phase lattice Boltzmann model and performed simulations on a digital rock model reconstructed from micro-CT scanned images. Single Relaxation Time (SRT) and Multi Relaxation Time (MRT) formulations of lattice Boltzmann Method (LBM) are also can used to simulate fluid flow through the pores in order to compute the permeability of porous media (Jithin et al., 2017). But both MRT and SRT showed over-prediction of the permeability compared to the experimental value, which could be the effect of an apparent slip at the solid surface due to a numerical error in the location of the wall.

Gharbi et al. studied the fluid-rock interactions during CO_2 storage in saline carbonate aquifers (Gharbi et al., 2013) using high resolution (8 μm) μ-CT. They extracted representative pore-throat networks to compute the average coordination number and assess changes in pore and throat size distributions, then compared pore structure and connectivity using image analysis and pore-scale modeling techniques. Pereira Nunes et al. found the new dissolution regime during the mineral dissolution by the injection of a CO_2-saturated brine (Pereira Nunes et al., 2016).

X-Ray μ-CT used together with physico-chemical measurements during experiments and fine-scale observations offers the opportunity to better access and understand reaction-induced changes of pore structure, permeability and reactivity surface, as a result of the interplay between geochemical reactions, changing rock geometry, and the hydrodynamic and transport properties (Noiriel, 2015). μ-CT is a method that allows registration and analysis of the internal structure of an object without violating its structure and integrity (Akasheva et al., 2020; Hounsfield, 1973).

This article discusses the impact of the acid flow rate on the permeability and porosity and pore structure of samples during experimental dissolution of carbonate core samples with a combination of X-Ray μ-CT. Aspects of processing 2D images of core samples obtained using X-Ray μ-CT are being studied.

Materials and methods. This chapter presents a description of considered carbonate core samples, and the procedure of solution injection and imaging using μ-CT.

Sample characterization. 8 carbonate core samples ~5 cm in length and ~3 cm in diameter were considered for the dissolution experiment in this work. The porosity and permeability of the samples range from 11.16-20.92% and 135.13-721.04 mD, respectively. Mineral composition of samples was determined using the X-ray diffractometer Bruker D2 Phaser. According to the results, samples 2, 9, 12, 13 and 18 consist of 100% calcite; samples 7, 10 and 11 consist of 99% calcite and 1% quartz. Thus, all studied samples are highly homogeneous in composition, which excludes the influence of mineral heterogeneity on acid-rock interaction.

Core sample's permeability was calculated using Darcy's equation , where is the permeability (m^2), is the dynamic viscosity (Pa·s), is the core sample length (m), is the volumetric flow rate of water (m^3/s), is the pressure drop at the ends of the core samples (Pa), is the sample cross-sectional area (m^2).

Injection of acid solutions. Dissolution experiments were conducted using 2% NaCl solution and solutions with 12% and 18% HCl concentrations. The experiments were performed at 20°C and 3 MPa. Solution injection was performed using Wille Geotechnik Y1000 (Fig. 1A). Acid breakthrough pore volume was calculated using the equation , where is the acid injected volume until breakthrough and is the sample's pore volume.

Imaging using X-ray computed tomography. Pre- and post-dissolution images of 8 dried samples were obtained using the General Electric phoenix v|tome|XS 240 X-Ray μ-CT (Fig. 1B). The method is based on the measurement and complex computer processing of the difference in the attenuation of X-ray radiation depending on the change in the density and atomic composition of the substance. The processing of obtained 2D images were done using Avizo® software in order to build a 3D rendered model of the samples and calculate macroscale properties such as porosity, permeability, specific surface area, etc.

In this study, μ-CT was used to identify lithological inhomogeneities, fracture zones, areas of localization of caverns and assess the structure of the pore space of core samples before and after dissolution.

Results and discussions. Measurements during the injection of acid solutions. Acid solutions with 12% (for samples 18, 9, 13 and 7) and 18% (for samples 12, 10, 11 and 2) HCl concentrations were injected into core samples at rates of 1, 2, 4, and 8 ml/min. The permeability ratio (final to initial) and acid breakthrough PVs as a function of flow rate are indicated in Figure 2.

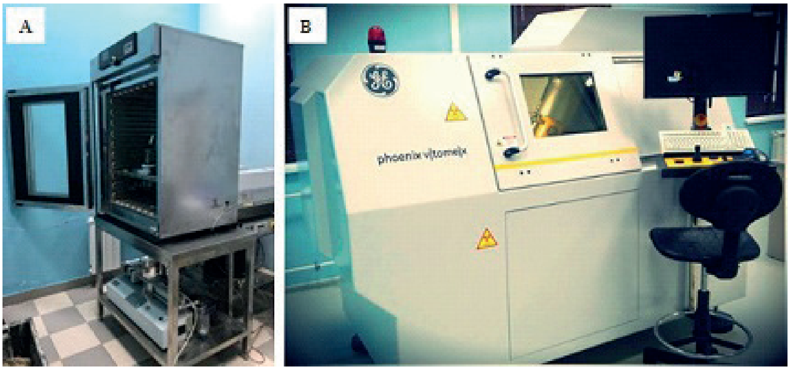

Figure 1. Flooding (A) and imaging equipment (B)

The permeability ratio and injected acid volume are important parameters when stimulating hydrocarbon inflow into a well. If it is possible to achieve a significant increase in permeability while consuming a minimal acid volume, then this will be the most optimal case for improving well productivity. According to Figure 2A, a significant increase in permeability was observed, especially at high (4 and 8 ml/min) flow rates for both 12% and 18% HCl concentrations. In the case of an injection of 18% HCl acid solution, although almost the same volumes of solution were injected at all flow rates (Fig. 2B), at low flow rates, the permeability ratio is already less than half of the ratio of high flow rates. Considering that samples 12, 10, 11 and 2 have almost the same porosity and different permeabilities, and the permeability of samples 11 and 2 is 1.5-2 times higher than that of samples 12 and 10 (see Fig. 2A), we can assume that there is a certain relationship between the permeability ratio and the initial permeability of the samples.

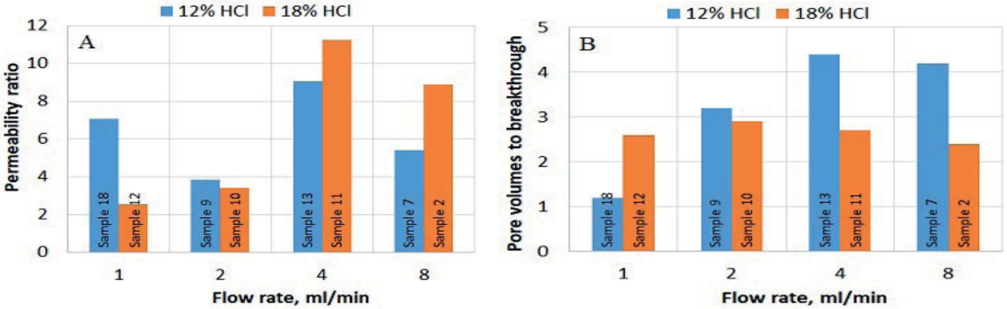

Figure 2. Permeability ratio (A) and breakthrough PVs (B) as a function of flow rate

Image processing. As a result of μ-CT scanning, 1700x1700x3000 slices were obtained for each of the 8 samples before and after acid injection. The voxel size is ~19 μm^3. These data are an essential starting point for image processing. Image processing was performed using the Avizo® software (https://www.fei.com/software/amira-avizo/.), and consisted of several steps, namely pre-processing and segmentation of each sample before and after acid solution injection.

Pre-processing includes cropping and filtering the images in order to remove noise. In fact, it is pre-processing that is an important part of the quantitative analysis of the main parameters of a porous media in a digital form. In addition, the next steps depend on the pre-processing, it must also be considered that the resolution of μ-CT images strongly affects the determination of key parameters of the sample material. Therefore, for example, Soulaine et al. show that for μ-CT images of sandstones with a resolution of 20 μm, it accurately determines porosity, and for carbonates, even about 1 μm is not enough to determine porosity. The fact is that in the images of carbonate-type samples, there are many pores that turn out to be blurry. These are also called pores at the sub-micron scale, which complicate circumstances when applying filters and further segmentation (Soulaine et al., 2016).

In this paper, for a noise reduction on the faces of samples, they were first cropped. After that, in order to select the appropriate filter, different filters were compared, namely: median, non-local mean, unsharp masking, as well as their combinations (Figure 3 and Table 1). More detailed information about these filters can be found in (Schlüter et al., 2014).

Segmentation in this paper lies in the separation of the pore space of the sample. Since the samples are almost carbonate rocks, only macropores (above 19 μm) were identified during the segmentation. A global segmentation method was used, in which the selection of pores is performed depending on the definition of the boundary of the intensity of visible pores.

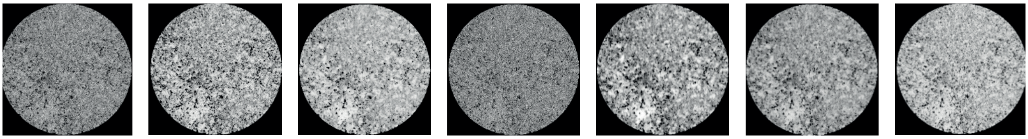
Original; Median; Non-Local Means; Unsharp Masking; Non-local +median; Non-local +unsharp; Median+ unsharp

Figure 3. Visual comparison of different filters on sample 7

Using of the unsharp masking filter gives non-physical porosity values at a several slices (Figure 4A). Combinations of different filters resulted in porosity reduction (Figure 4A, Table 1). Since the image porosity value at the resolution of 19 μm and above gave a worse match with measured values, the focus of selecting a suitable filter was to cover more pores. Based on this, the choice fell on the median filter, since the median filter covers more pores (Figure 4A) and deviation from the measured porosity is minimal in comparison with the rest (Table 1). The non-local means filter at this resolution showed a decrease in porosity, since there were a lot of blurry areas, and it is also very computer resource-intensive. However, at very high resolutions (~1 μm), the non-local means filter performs well in most cases.

Since it was not possible to cover the full range of pores, the total image porosity of the samples did not coincide with their measured porosity. Saxena et al. introduced a correction factor in order to match the image porosity with the experimental one as , where – image porosity, and – measured porosity (Saxena et al., 2019). The values of correction coefficient for considered core samples are shown in Figure 4 B. According to the values of the correction coefficient for all samples, it was observed that the image porosity deviated from the experimental porosity value by almost 35-65% due to the low resolution of μ-CT. This confirms that for carbonate samples, the resolution of about ~20 microns are insufficient to capture the entire pore space.

Table 1 – Image porosity of the sample 7 obtained by different filters

Filter Name	Image porosity, %	100*(Filtered-Measured)/Measured, %
Median	13,49	-35,4
Non-Local Means	7,61	-63,5
Unsharp Masking	11,66	-44,2
Median+Non-Local Means	7,83	-62,5
Median+Unsharp Masking	12,96	-38,0
Non-Local Means+Unsharp Masking	6,65	-68,1

Pore structure change due to dissolution. The μ-CT was used to construct a 3D model of the samples in order to study the changes in the pore structure and to calculate macroscopic parameters before and after dissolution. Based on the μ-CT, an assessment of changes in the structure of the pore space of samples before and after dissolution was carried out (Figure 5).

Figure 5 demonstrates the peculiarities of the action mechanisms of solutions on the carbonate cores. It has been noted that the acid solution was injected from top to bottom and wormholes are indicated as a dark-blue region (lower pictures in Figure 5). As it can be seen, for all samples, a slight dissolution of the inlet face is observed, which is associated with the high dissolving rate of the solutions used. The dissolution pattern was conical for low flow rates (1, 2 ml/min), but for high (4, 8 ml/min) flow rates it becomes more dominant for both 12% and 18% HCl concentrations (Figure 5). For sample 18, the dissolution front developed diagonally from right to the left due to the natural fracture. It can also be noted, that with an increase of the flow rate, the dominant wormhole begins to develop and thicken.

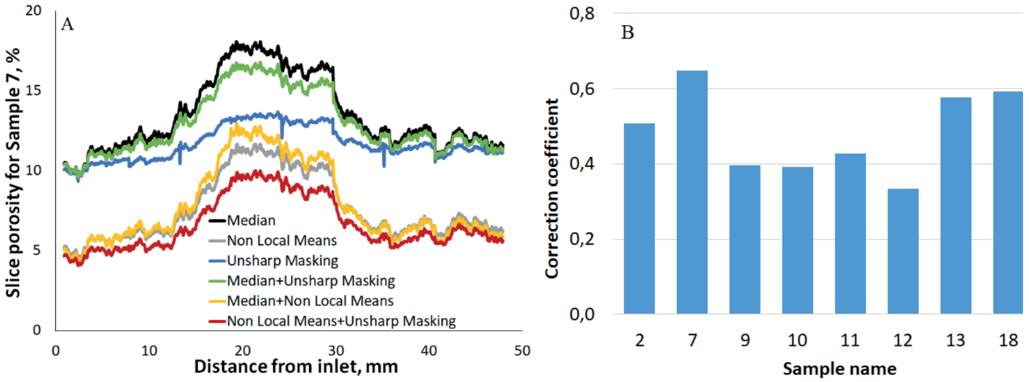

Figure 4. Slice averaged image porosity of the sample 7 obtained by different filters versus distance (A) and the value of the correction coefficient for different samples (B)

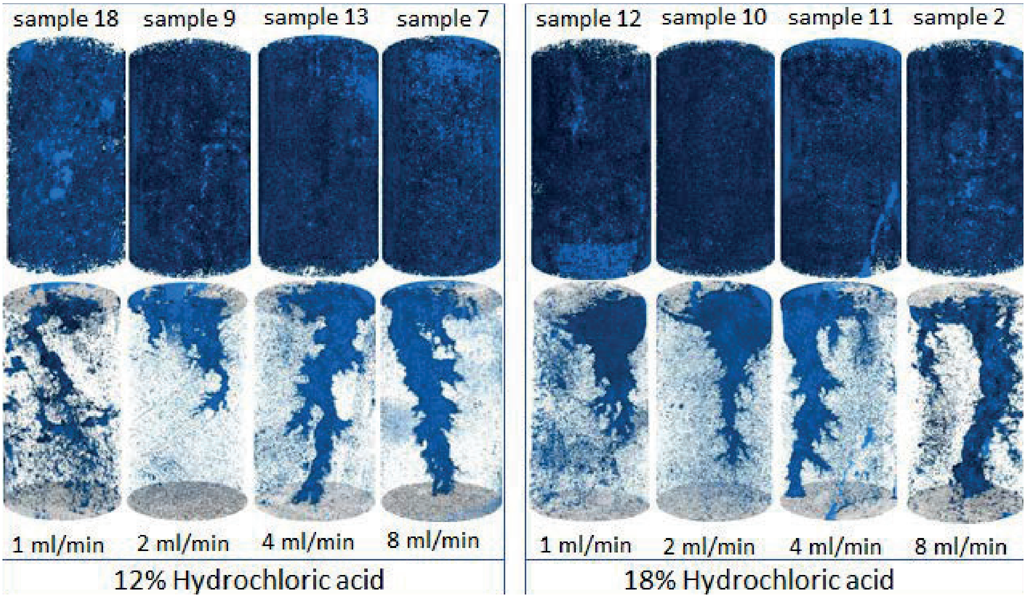

Figure 5. 3D models of samples before (upper) and after (lower) dissolution versus flow rate

The microcomputed tomography used in this work helped to calculate the slice-averaged porosity and this allows us to see the heterogeneities in the porosity along the sample length. The slice-averaged porosity distribution along the length of the sample is shown in Figure 6. In Figure 6, the graphs in the upper and lower rows correspond to 12% and 18% HCl concentrations, and the columns from left to right correspond to different flow rates of the solution, while dashed and solid lines show the distributions of porosity before and after dissolution. As it can be seen from Figure 6, low flow rates (1 and 2 ml/min) lead to intensive dissolution of the rock mainly in the inlet part of the samples, especially when 12% hydrochloric acid is injected. On the contrary, at high flow rates (4 and 8 ml/min), almost uniform dissolution of the rock is observed along the length of the samples, indicating that dominant wormholes were formed, and further injection of the acid solution contributed to their widening. This effect can also be seen for lower flow rates (1 and 2 ml/min) in the case of solution injection with 18% HCl concentration. This means that rock dissolution is a complex process characterized by competition between solution transport and acid-rock interaction, with varying degrees of heterogeneity in pore distribution and permeability. All this leads to different rates of dissolution of the carbonate rock, although the rocks have almost the same characteristics. This is better seen in Figure 7.

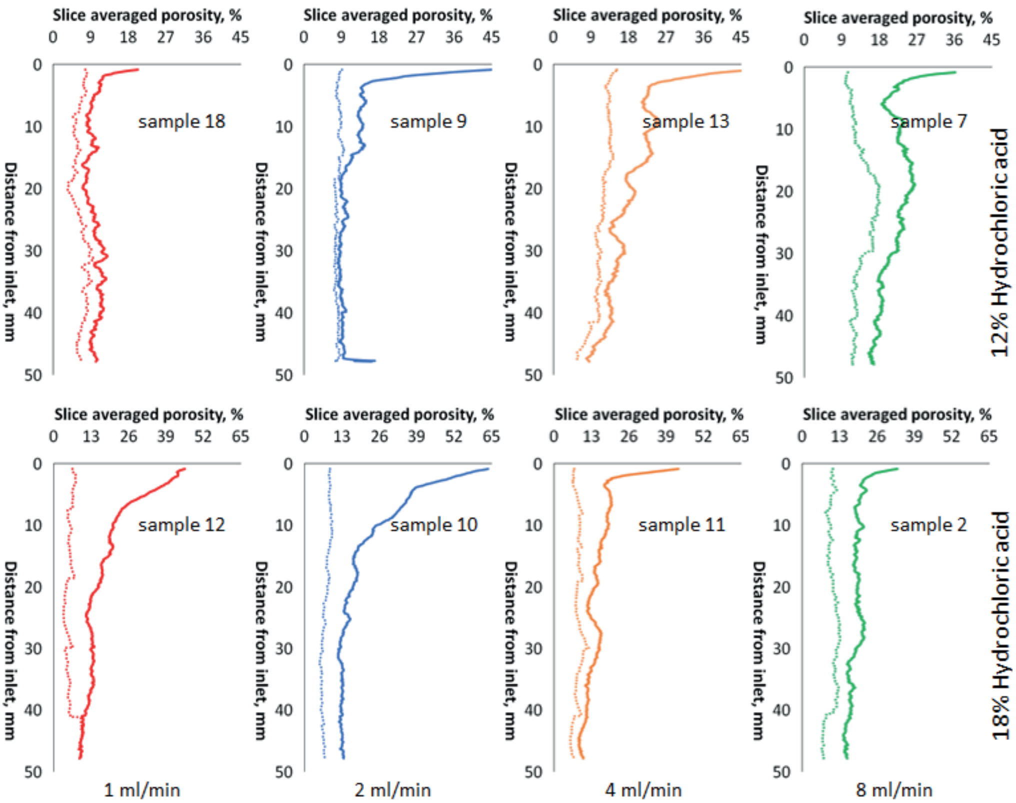

Figure 6. Slice averaged porosity distribution along the sample length for 12% HCl (Upper layer) and 18% HCl concentrations (Lower layer) at different flow rates (Columns)

The change rate of the sample-scale porosity in time is shown in Figure 7 as a function of solution flow rate.

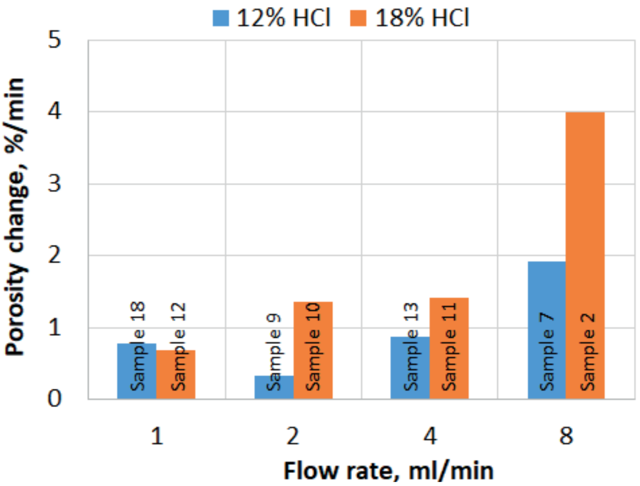

Figure 7. Porosity change rate versus flow rate for 12% HCl and 18% HCl concentrations

The change rate of sample-scale porosity in time is calculated as the ratio of the difference in sample-averaged porosity after and before the solution injection to the time of rock dissolution before acid breakthrough. In Figure 7, there is a clear relationship between the change rate of porosity and the flow rate of the solution – with an increase in the flow rate, the change rate of porosity increases. This is very noticeable in the case of a solution injection with an 18% HCl concentration, although almost the same volume of acid was injected at all rates (see Figure 2B). This may be related to the initial permeability of samples 12, 10, 11 and 2. It can also be seen that the change rate of porosity is in good agreement with the permeability ratio (see Figure 2A) in the case when a solution with 18% HCl concentration is injected. On the contrary, when a solution with 12% HCl concentration is injected, this relationship is not obvious, which may indicate that the absolute permeability of a porous medium depends not only on porosity, but also on other factors, such as pore connectivity and specific surface area.

Conclusion. In this work a combination of experimental and imaging techniques were applied to study the interaction of carbonate samples with the acid solutions. All studied samples are almost completely calcite, which minimizes the influence of the mineralogy heterogeneity on the dissolution.

According to the results, a significant increase in permeability due to dissolution was observed.

The processing of images obtained using μ-CT show that the resolution of about 19 μm is not enough to estimate the overall porosity of the samples. Also, it has been shown that at this resolution the median filter is the most suitable. Using this filter, it was observed that the image porosity is 35-65% of the experimental one depending on the samples.

The change of the 3D pore structure due to dissolution shows that the dissolution pattern was conical for low flow rates (1 and 2 ml/min), but for high (4 and 8 ml/min) flow rates it becomes more dominant for both 12% and 18% HCl concentration. Further injection of both 12% and 18% HCl concentrations at high rates (4 and 8 ml/min) after the dominant wormholes formed contributed to their widening. This effect can also be seen for lower flow rates (1 and 2 ml/min) in the case of a solution injection with 18% HCl concentration.

It has been observed that the flow rate significantly impacts the sample-averaged dissolution rate. With an increase in the flow rate, the sample-averaged dissolution rate increases.

Further work will include a study of the effect of the flow rate on the fluid flow in a porous medium using pore-scale modeling. Also, the applicability of an empirical equation for calculating the absolute permeability in the case of a 3D pore structure change of the samples during the dissolution will be studied.

***ACKNOWLEDGMENT.** This research is funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan (Grant No. AP08052055 and AP09058419), which is gratefully acknowledged by the authors.*

Z. Akasheva also expresses her gratitude to the PhD grant program organized by WEF (Chevron, Eni and Shell) for the opportunity to undertake a foreign internship.

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ISSN 2518-170X (Online),
ISSN 2224-5278 (Print)

Заместитель директор отдела издания научных журналов НАН РК *Р. Жәлиқызы*
Редакторы: *М.С. Ахметова, Д.С. Аленов*
Верстка на компьютере *Г.Д.Жадыранова*

Подписано в печать 18.02.2023.
Формат 70x90$^1/_{16}$. Бумага офсетная. Печать – ризограф.
20,0 п.л. Тираж 300. Заказ 1.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-19