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Magnetic properties of lake Kandrykul sediments (Republic of Bashkortostan, Russian Federation)

D.M. Kuzina✉, A.R. Yusupova, N.G. Nurgalieva, D.K. Nurgaliev, P.S. Krylov, D.I. Mulikova

Kazan Federal University, Kazan, Russian Federation

✉di.kuzina@gmail.com

Abstract. Relevance. It is known that sedimentary deposits in modern lakes with high sedimentation rates contain detailed paleoclimatic, paleobiological, paleoecological and magnetic records. The study of these parameters allow creation of paleoreconstructions and prediction of future climate behavior. **Aim.** To identify ecological and climatic events and trends during the Late Quaternary, based on magnetic properties of rocks. Some climate-dependent parameters are not properly used, we are showing that magnetic parameters are sensitive indicators of climate change. **Object.** Sediments of the Lake Kandrykul (Republic of Bashkortostan). **Materials and methods.** A set of petromagnetic studies was carried out, including measurements of magnetic susceptibility, normal remanent magnetization in external magnetic field of up to 1.5 T, as well as differential thermomagnetic analysis on induced magnetization and scanning electron microscopy on selected samples. Normal magnetization curves were used to set apart dia-/paramagnetic, ferromagnetic and superparamagnetic components. **Results.** Seismoacoustic studies showed that the lake bed is smooth, without sharp changes. The maximum water depth is about 16 m, the thickness of sediments reaches 7.5 m. Magnetic susceptibility was measured for three core columns, and the results show good correlation of this parameter between all the cores. Thermomagnetic analysis was used to determine magnetic minerals composition. The Day–Dunlop plot showed the presence of single, pseudo-single and multidomain grains. The authors determined the contribution of dia/para-, ferro-, and superparamagnetic components to the overall magnetic susceptibility. The paper considers the variations in magnetic susceptibility, para-, ferromagnetic components in the context of the Holocene climate changes. **Conclusions.** Variations in magnetic properties reflect climate changes (i.e. deposition environment). Six zones with different depositional conditions were distinguished based on magnetic susceptibility and its component changes. Additionally, zones with single-domain particles were highlighted based on hysteresis parameters variations. These zones most likely are associated with the remains of magnetotactic bacteria. Joint analysis of paramagnetic, ferromagnetic susceptibility and hysteresis parameters produced information on the effect of total humidity on the bioproductivity of the lake.

Keywords: lake sediments, magnetic properties, hysteresis parameters, biogenic magnetic grains, paleoclimate, paleoenvironment reconstruction

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Магнитные свойства донных отложений озера Кандрыкуль (Республика Башкортостан, Россия)

Д.М. Кузина✉, А.Р. Юсупова, Н.Г. Нургалиева, Д.К. Нургалиев, П.С. Крылов, Д.И. Муликова

Казанский федеральный университет, Россия, г. Казань

✉di.kuzina@gmail.com

Аннотация. Актуальность. Известно, что осадочные отложения в современных озерах с высокой скоростью седиментации содержат детальные палеоклиматические, палеобиологические, палеоэкологические и магнитные записи. Изучение этих данных позволяет создавать палеореконструкции и прогнозировать поведение климата в будущем. **Цель:** выявление экологических и климатических событий и тенденций в позднечетвертичное время на основе магнитных свойств горных пород. Некоторые климатозависимые параметры не используются должным образом, в данной работе показано, что магнитные параметры являются чувствительным индикатором изменения климата. **Объект:** озерные отложения из керна озера Кандрыкуль, Республика Башкортостан. **Методы.** Проведен комплекс петромагнитных исследований, включающий: измерение магнитной восприимчивости образцов трех керновых колонок; получение кривых нормального остаточного намагничивания при непрерывном росте внешнего магнитного поля до 1,5 Тл для одной колонки; дифференциальный термомагнитный анализ по индуцированной намагниченности и сканирующую электронную микроскопию по выборочным образцам. По кривым нормального намагничивания было проведено разделение магнитной составляющей на диа-/парамагнитную, ферромагнитную и суперпарамагнитную компоненты. **Результаты.** Сейсмоакустические исследования показали, что дно озера ровное, без резких перепадов. Максимальная глубина озера составляет около 16 м, мощность осадочных отложений достигает 7,5 м. Магнитная восприимчивость измерялась по трем кернам, результаты показали хорошие вариации этого параметра по всем кернам. Это позволяет коррелировать керны между собой. Термомагнитный анализ позволил определить состав магнитных минералов. По данным коэрцитивной спектрометрии построена диаграмам Дэя–Данлопа, которая показывает наличие в осадках однодоменных, псевдооднодоменных и многодоменных зерен. Определена роль диа-/пара-, ферро- и суперпарамагнитных компонентов в общей магнитной восприимчивости. Вариации магнитной восприимчивости, пара- и ферромагнитной составляющих рассмотрены в контексте голоценовых климатических изменений. **Выводы.** Изменения магнитных свойств могут быть использованы для описания изменений климата (экологических и седиментационных условий). На основе вариаций магнитной восприимчивости и ее составляющих выделено шесть зон с различными условиями осадконакопления. Кроме того, по изменениям параметров гистерезиса выделены зоны с однодоменными частицами, которые, вероятнее всего, связаны с наличием в осадке остатков магнитотактических бактерий. Совместный анализ парамагнитной, ферромагнитной восприимчивости и параметров гистерезиса позволил получить информацию о влиянии влажности на биопродуктивность озера.

Ключевые слова: озерные отложения, магнитные свойства, гистерезисные параметры, биогенные магнитные зерна, палеоклимат, реконструкция палеосреды

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Introduction

It is known that sedimentary deposits in modern lakes with high sedimentation rates contain detailed paleoclimatic, paleobiological, paleoecological, and magnetic records [1]. The diversity of lake types and numerous factors that determine sedimentation ensure unique qualitative and quantitative composition of the sediment in each individual lake. The study of these processes allow creation of paleoreconstructions and prediction of future climate behavior [2–4].

There are many indirect data types that can be used for paleoreconstructions, such as geochemical, isotopic, and paleobiological indicators. Magnetic properties of sediments, on the other hand, are not so widely used for this task. Nevertheless, changes in magnetic properties that reflect the supply of sedimentary material into the lake can be used to reconstruct the climate history and the environment in the Quaternary.

Magnetic properties reflect concentration, mineralogy and grain size of the magnetic components in sediments, which are often affected by physical, chemical and biological processes [5–7]. Thus, deep understanding of the processes that affect magnetic properties is crucial to reliable interpretation of results of any paleoenvironmental and paleoclimatic studies [1, 6, 7].

The aforementioned magnetic properties can be studied through measurements of magnetic susceptibility (MS), coercive spectrometry, and differential thermomagnetic analysis (DTMA). MS reflects changes in the supply of clastic material from water or eolian sources [8].

MS of rocks and sediments depends on the mineral composition and structure of magnetic domains. Therefore, different rocks and sediments show different MS, and many environmental studies note lithology impact on magnetic properties [9–12].

Normal magnetization curves can be used to determine hysteresis parameters, the domain structure and the size of ferrimagnetic grains [13], and the contribution of para-, ferro- and superparamagnetic components to the induced magnetization and MS [5]. The paramagnetic component reflects the inflow of allothigenic material into the sedimentation basin, which, in its turn, correlates with humidity and water levels. The ferromagnetic component is often represented by single domain (SD) biogenic (authigenic) grains, as well as multidomain (MD) clastic material of allothigenic nature.

The results of previous petro- and paleomagnetic studies of Lake Kandrykul sediments were published in [14, 15].

This paper focuses on the 5.4 m long core column extracted from Lake Kandrykul in 2021. To study the climate changes within the research area, thermomagnetic analysis was carried out, accompanied by measurements of MS and evaluation of hysteresis parameters. A review of already performed regional studies shows that detailed magnetic studies of lake sediments can supplement and expand the paleoclimatic data in the study area.

Materials and methods

The main aim of this work is to use magnetic properties of lake sediments in the study of ecological and climatic events and trends during the Late Quaternary. To achieve the aim, magnetic measurements were performed, and the results were processed and interpreted.

The object of investigations, Lake Kandrykul, is located in the Republic of Bashkortostan (54°30′10″ N, 54°03′50″ E; the Tuymazinsky district, Russia) in the forest-steppe zone of the Volga-Kama basin [16]. It lies within the territory of the Nature Park of the same name and belongs to the basin of the Ik River. The size of the lake is 6.55×2.38 km (15.6 km²), the maximum depth is 16.5 m, and the average depth is 7.2 m [17]. The lake is of karst origin [16, 18]. The water is mildly alkaline (pH~8.7) and has hydrocarbonate-sulfate sodium-magnesium composition, type II [16, 19].

High-resolution seismic reflection survey of the lake area was conducted using 1.5 kHz Boomer source along six profiles (Fig. 1).

Based on seismoacoustic studies and lithologic description, three core columns with a length of 378–524 cm were extracted. The total number of core samples was 659 with a sampling step of 2 cm along the core. Core column #3 was chosen for a detailed analysis.

The analysis included MS measurements, coercive spectrometry, DTMA, determination of mineral and chemical composition.

MS was measured using the Multifunction Kappabridge MFK1-FA (AGICO). The measurements were carried out at a standard frequency of 976 Hz [20].

A coercive spectrometer (*J_meter*) was used to study the lake sediment in terms of hysteresis parameters [21, 22]. The hysteresis parameters made it possible to separate measured remanent and induced magnetization in magnetic fields of up to 1.5 T at room temperature.

In this way, remanent saturation magnetization (Mrs), saturation magnetization without the paramagnetic component (Ms), coercive force without the paramagnetic component (Bc), and remanent coercive force (Bcr) were evaluated. These parameters were used to determine the state of magnetic domains, as well as the grain size of ferromagnetic minerals. The magnetic hysteresis parameters reflect the contribution of the paramagnetic (k_para), ferromagnetic (k_ferro), and superparamagnetic (k_super) components to the total MS [23, 24]. The primary source of k_para is most probably allothigenic material, while k_ferro and k_super components can be of a mixed (allothigenic and/or authigenic) nature [23, 24].

Thermomagnetic analysis is often used for studying the ferromagnetic fraction in rock samples [21, 25]. DTMA of the samples uniformly distributed along the studied section was carried out using a Curie balance. Each sample taken from core column #3 was heated twice in a magnetic field of 0.4 T at a heating rate of 100°C/min. The result was the temperature dependences of the induced magnetization Ji(T) obtained during the first and second heating.

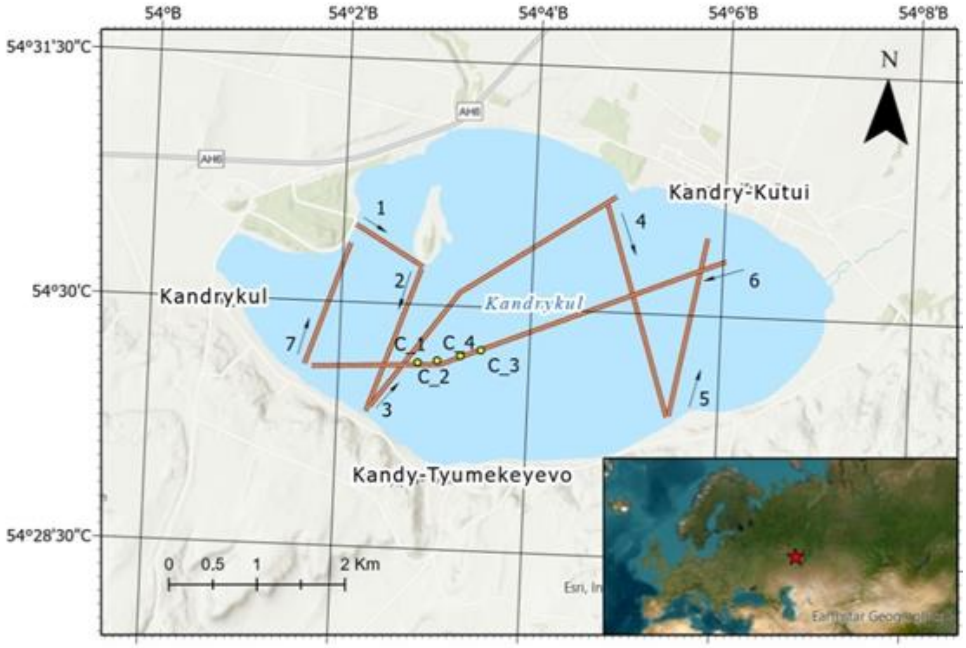


Fig. 1. *Location of Lake Kandrykul. Red lines indicate seismoacoustic profiles, red dots mark positions of core sampling places*

Рис. 1. *Расположение озера Кандрыкуль. Красной линией показаны сейсмоакустические профили, красными точками отмечены места отбора керна*

Electron microscopy was carried out for several samples. Measurements are made at the Interdisciplinary Center for Analytical Microscopy (Kazan Federal University) on Carl Zeiss "Merlin" field emission scanning electron microscope equipped with the Aztec X-MAX (for elemental analysis) detector. The resolution was 127 eV. Sample preparation for measurements consisted of grinding and etching organic matter.

Results

Seismoacoustic studies showed that the lake bed is smooth, without any sharp changes in its terrain (at least along the profiles). The maximum water depth in the lake is about 16 m, and the thickness of sedimentary deposits reaches 7.5 m. Based on the results of preliminary studies along the sixth profile (Fig. 2), four core columns were taken from the place with no gas and undisturbed stratification.

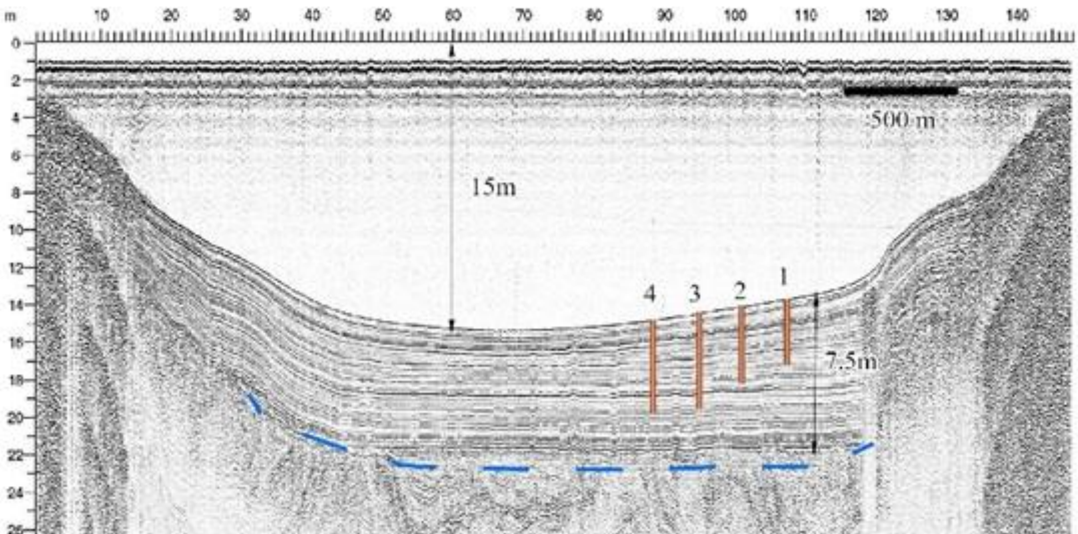


Fig. 2. *Seismic profile #6. Blue dotted line marks the time of the lake basin formation. Red lines depict core columns*

Рис. 2. *Сейсмоакустический профиль #6. Синей пунктирной линией отмечено время образования озерного бассейна. Красной линией изображены керновые колонки*

Fig. 3 presents the results of MS measurements. MS values vary in the ranges of (0.56–21.94)*10^{-7} m^3/kg, (0.54–16.98)*10^{-7} m^3/kg, and (0.58–16.94)*10^{-7} m^3/kg for core columns #1, #2, and #3, respectively.

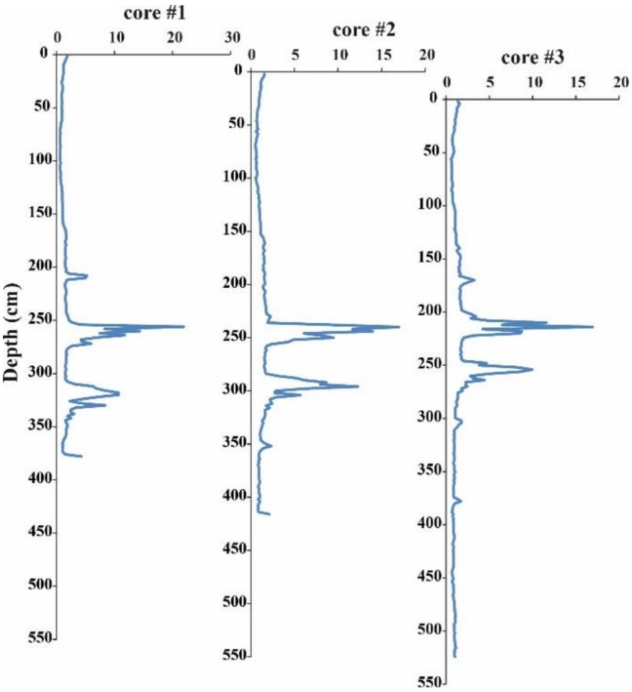


Fig. 3. *Variation of MS (*10⁻⁷ m³/kg) with depth*
Рис. 3. *Вариации магнитной восприимчивости по глубине (·10⁻⁷ м³/кг)*

The Day–Dunlop plot [10, 13] (Fig. 4) obtained with the *J_meter* spectrometer shows the magnetic hysteresis parameters for core column #3.

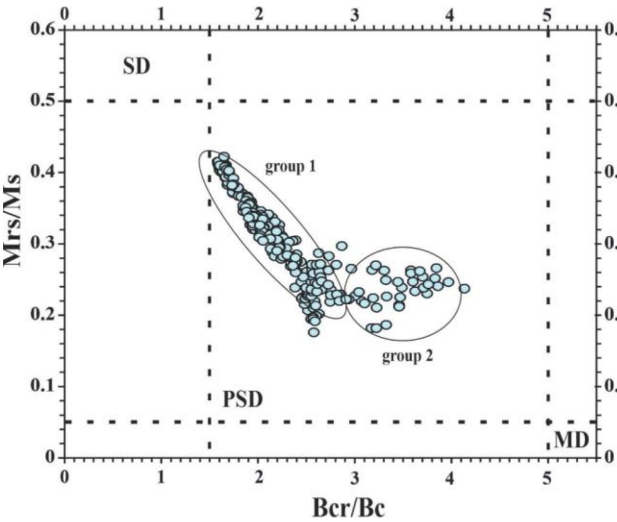


Fig. 4. *Day–Dunlop plot [13, 26] for samples taken from core column #3*
Рис. 4. *Диаграмма Дэя–Данлопа [13, 26] для образцов керновой колонки #3*

Based on the hysteresis parameters (M_{rs}/M_s and B_{cr}/B_c), the samples can be divided into two groups. Group #1 includes samples, in which the magnetic component is represented by a mixture of SD and MD or pseudo-single domain grains (PSD) [13]. The larger the M_{rs}/M_s ratio and the smaller the B_{cr}/B_c ratio, the more SD grains are contained in the sample. The presence of SD grains in the sediments of modern lakes means the presence of biogenic magnetic grains [15]; therefore, it can be concluded that the magnetic component of Group #1 samples is represented by a mixture of SD biogenic (authigenic) grains and MD magnetic grains, most likely brought into the sedimentation basin. Samples of Group #2 have a more complex composition: they contain few SD grains (of biogenic origin); most of the magnetic grains are represented by MD and superparamagnetic (SP) grains. These two groups of sediments should have formed under different environmental conditions. The samples of the first group represent sediments formed under conditions of high bioproductivity, probably in a warm and humid climate. The samples of the second group were formed under conditions of low bioproductivity. These data are very useful for paleoenvironmental reconstructions, which are discussed below.

The MS components identified while studying core column #3 samples change insignificantly with depth (Fig. 5). It can be seen from k_full that the values change in the range of (5.24–166.02)*10^{-5}; k_para varies from 3.31*10^{-5} to 7.72*10^{-5}; k_ferro values range from 1.59*10^{-5} to 156.93*10^{-5}; k_super values vary from 0.03*10^{-5} to 2.45*10^{-5}.

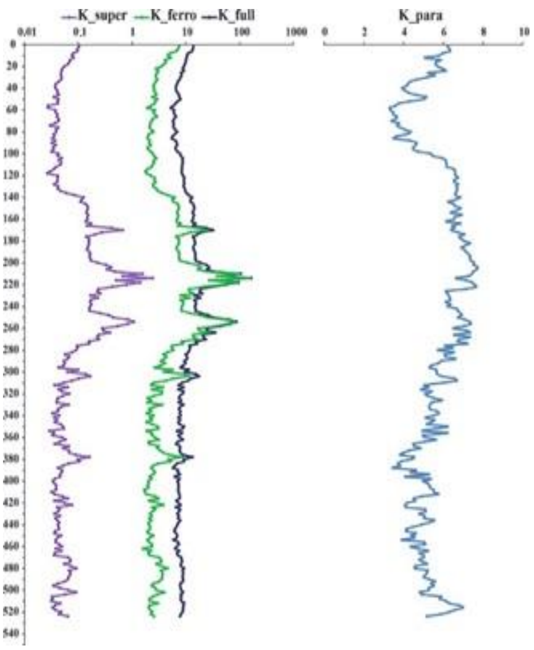
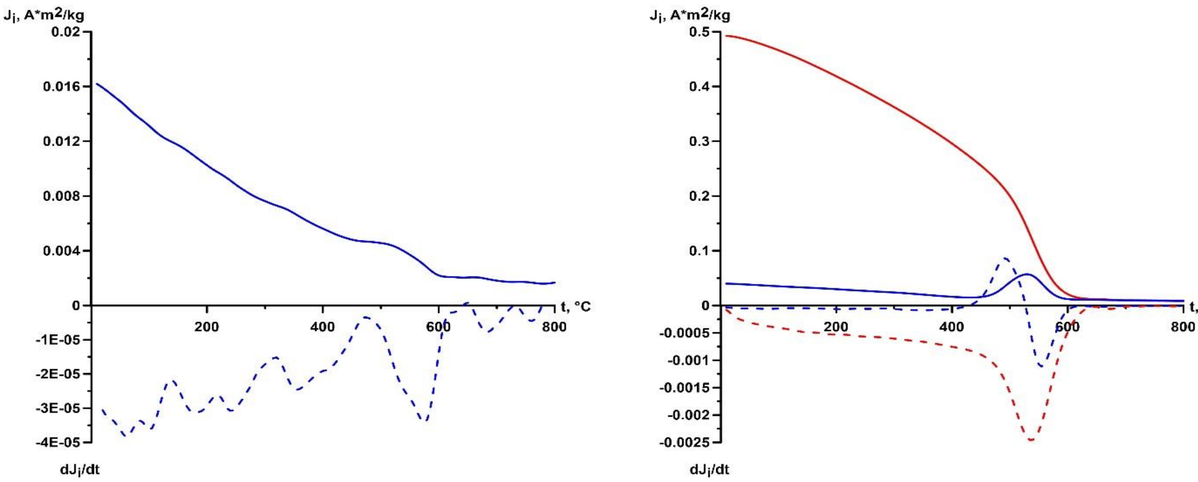


Fig. 5. *Variations of magnetic parameters obtained for core sample #3*
Рис. 5. *Вариации магнитных параметров образцов керновой колонки #3*

Differential thermomagnetic analysis showed that magnetic minerals in the sediment are represented by magnetite (Fig. 6, *a*) and iron sulfides (Fig. 6, *b*).

There is a difference between the top and bottom halves of the core. Magnetite is present in the first half of the core; it can be seen on the first heating curve, with the Curie temperature around 580°C (Fig. 6, *a*). Reducing conditions occur due to the combustion of organic material, which contributes to the formation of new magnetic minerals. It leads to the increase in magnetization after the first heating. The second half of the core contains iron sulfides (an increase in the magnetization around 450°C), which, when heated, transform into a more magnetic mineral, magnetite (Fig. 6, *b*).

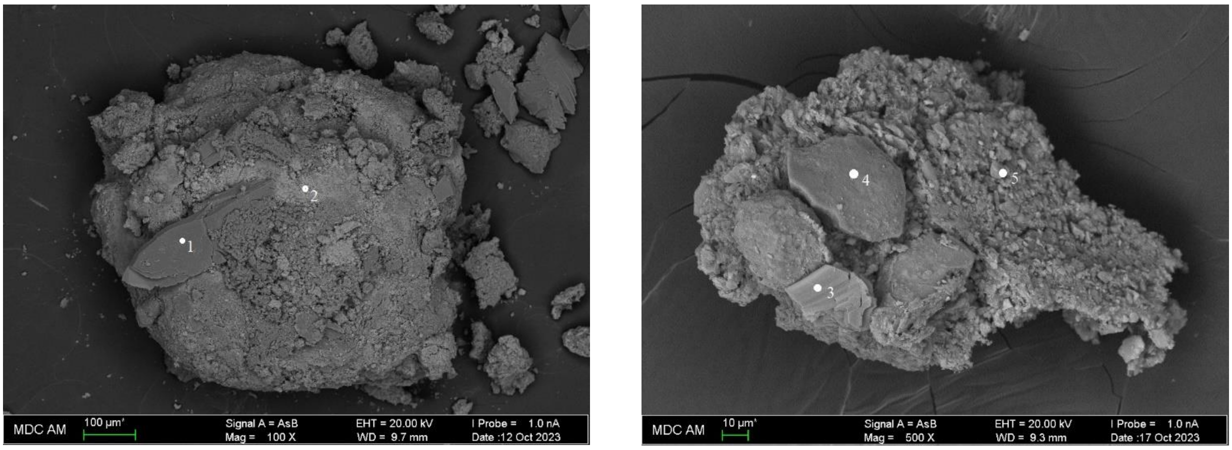
Results of electron microscopy shows the presence of different minerals in sediments. Magnetic minerals are presented mostly by iron oxides and silicates, also there are a few iron sulfides. These results correspond with DTMA results. The rest of material presented by different silicates, gypsum (in a big amount), dolomites, calcites, diatoms etc. (Fig. 7). Magnetic minerals are a bit etched, it can be because of organic matter etching.



a *b*

Fig. 6. *DTMA results for the samples taken from core column #3: a) #541 (82 cm); b) #651 (302 cm). Blue line represents the first heating, red line is for the second heating; solid lines are integral curves, dotted lines are differential curves*

Рис. 6. *Результаты дифференциального термомагнитного анализа для образца керновой колонки #3: а) #541 (82 см;) b) #651 (302 см). Сплошная синяя линия – первый нагрев, сплошная красная – второй нагрев; пунктирная синяя линия – дифференциал первого нагрева, пунктирная красная – дифференциал второго нагрева*



a *b*

Fig. 7. *Electron microscopy images of samples 607 (a) and 632 (b) in depths 114 and 164 cm respectively. All numbers in atom % and given only for elements with more than 1%. 1) O –69.21; Ca – 26.88; S – 15.66; 2) O – 55.16; C – 23.61; Fe – 17.39; S – 2.56; 3) O – 57.57; C – 21.21; S – 8.80; Ca – 7.99; Si – 2.97; 4) C – 60.36; Si – 17.91; O – 16.39; Fe – 2.06; 5) O – 52.02; C – 20.71; Si – 14.32; Al – 5.37; Na – 3.53; Ca – 2.02*

Рис. 7. *Электронно-микроскопические снимки образцов 607 (a) и 632 (b) на глубинах 114 и 164 см соответственно. Числа даны в атом. % для элементов, содержание которых превышает 1 %. 1) O – 69,21; Ca – 26,88; S – 15,66; 2) O – 55,16; C – 23,61; Fe – 17,39; S – 2,56; 3) O – 57,57; C – 21,21; S – 8,80; Ca – 7,99; Si – 2,97; 4) C – 60,36; Si – 17,91; O – 16,39; Fe – 2,06; 5) O – 52,02; C – 20,71; Si – 14,32; Al – 5,37; Na – 3,53; Ca – 2,02*

Discussion

Magnetic properties of sediments can be used to keep track of lithogenic input into the sedimentation basin, and can therefore serve as records of past environmental changes [27]. Variations in the concentration of magnetic minerals provide information on export of terrigenous sediments from land [11, 28, 29]. MS and hysteresis parameters (M_{rs}/M_s, B_{cr}/B_c) are quite informative and complement each other when being analyzed together in a study of modern lake sediments [30]. MS is useful for correlation of core samples taken from a single lake [1], as shown in Fig. 3. Since the core columns were taken from different parts of the lake bed, they have different length and features associated with different sedimentation rates and erosion caused by currents in different parts of the lake.

In our case, the analysis was based on k_para and k_ferro, which carry information about various components of the magnetic fraction. These components have different origin, and variations in their content in the sediment carry information about various factors that define the sedimentary environment [23, 24]. An increase in the paramagnetic component indicates an increase in the input of terrigenous material, which means more humid climate [1, 23, 24].

Variations in the composition of magnetic mineral assemblages can also be used for detecting changes in terrestrial climatic conditions, e. g., weathering and soil formation [29, 31–33]. Ferromagnetic susceptibility (k_ferro) depends on the content of ferromagnetic (ferrimagnetic) minerals in the sediment [1, 23, 24]. These minerals may be transported into the sedimentation basin from the surrounding catchment area, but may also be of biogenic nature [27, 34]. Magnetotactic bacteria living in the oxic/anoxic transition zone in the topmost part of the sediment utilize magnetic minerals, and can produce either magnetite or greigite intracellularly [34–37]. Furthermore, iron-reducing bacteria may induce the authigenic precipitation of magnetite [35, 38]. Most of the ferrimagnetic minerals (magnetite and sulfides) in the sediments of Lake Kandrykul are of biogenic nature. Hysteresis parameters (M_{rs}/M_s, B_{cr}/B_c) allow estimation of magnetic grain size through determination of their domain structure. SD grains have a very narrow size range of about 50–100 nm, and their presence in the sediments of modern lakes mean that they are most probably of biogenic nature [23]. Magnetotactic bacteria (MTB) that produce SD magnetic grains live at the border of the oxic/anoxic transition zone (OATZ) [34–36, 39]. The higher this zone located in the water column, the higher the bioproductivity of the lake. With low bioproductivity, OATZ goes deeper into the sediment, and the production of biogenic magnetic grains decreases or stops. It was assumed that the epochs of high bioproductivity and high content of biogenic magnetic grains in the sediment are associated with climatic optima (elevated average air temperatures) against the background of a very high rate of water inflow into the sedimentation basin.

Fig. 8 shows the parameters required for the analysis. Superparamagnetic susceptibility was not included, since it correlates very strongly with k_ferro, which is due to the presence of superparamagnetic grains smaller than SD, also of biological origin.

First of all, it should be noted that the samples in groups #1 and #2 (Fig. 4) are confined to different parts of the sediment. Samples of group #2 were taken from the upper part of the sediment (0–164 cm), and also appear in two more intervals of the section: (176–198 cm) and (234–244 cm). It can be said that the bioproductivity of the lake sharply decreased in these intervals. The sections of the core, in which the content of biogenic magnetic grains sharply increases, are marked with gray colored zones (*a*–*g*) in Fig. 8. In these zones, the M_{rs}/M_s ratio increases, while the B_{cr}/B_c ratio decreases, which indicates an increase in the number of SD grains of biogenic nature [40]. These zones are also reflected in the k_para variation curve. Zones *a*, *e*, *f*, in which the content of biogenic magnetic grains is not the highest, are marked by a decrease in k_para, which indicates a decrease in water supply to the sedimentation basin and the beginning of eutrophication of the lake with climate warming. Zones *b*, *c*, *d* are marked by some increase in k_para, which may indicate an increase in water inflow and an increase in the bioproductivity of the lake. Zones *b*, *c* are anomalous in terms of the content of biogenic magnetic grains. Between zones *b* and *c* there is a deposit section, in which the content of SD grains sharply decreases and this correlates with a sharp decrease in k_para. At different moments in time, the lake reacts differently to an increase in humidity and changes in water inflow into the basin. It depends on the average temperature.

Six zones (I–VI) with different sedimentation conditions can be distinguished based on magnetic susceptibility and its components.

Zone I (0–28 cm) is characterized by an increase in the ferrimagnetic component. The hysteresis parameters imply that this increase is due to MD and SP grains. The paramagnetic component of MS increases as well, which indicates an increase in terrigenous input and water inflow into the basin.

Zone II (28–114 cm) is characterized by a major drop of k_para. It is possible that the water level dropped at that time, and the lake was fed by groundwater. Gypsum and anhydrite of Permian age are widespread in the study area [41], which perhaps led to the inflow of sulfates into the basin, and MB began to produce magnetic iron sulfides (greigite?) (Fig. 6).

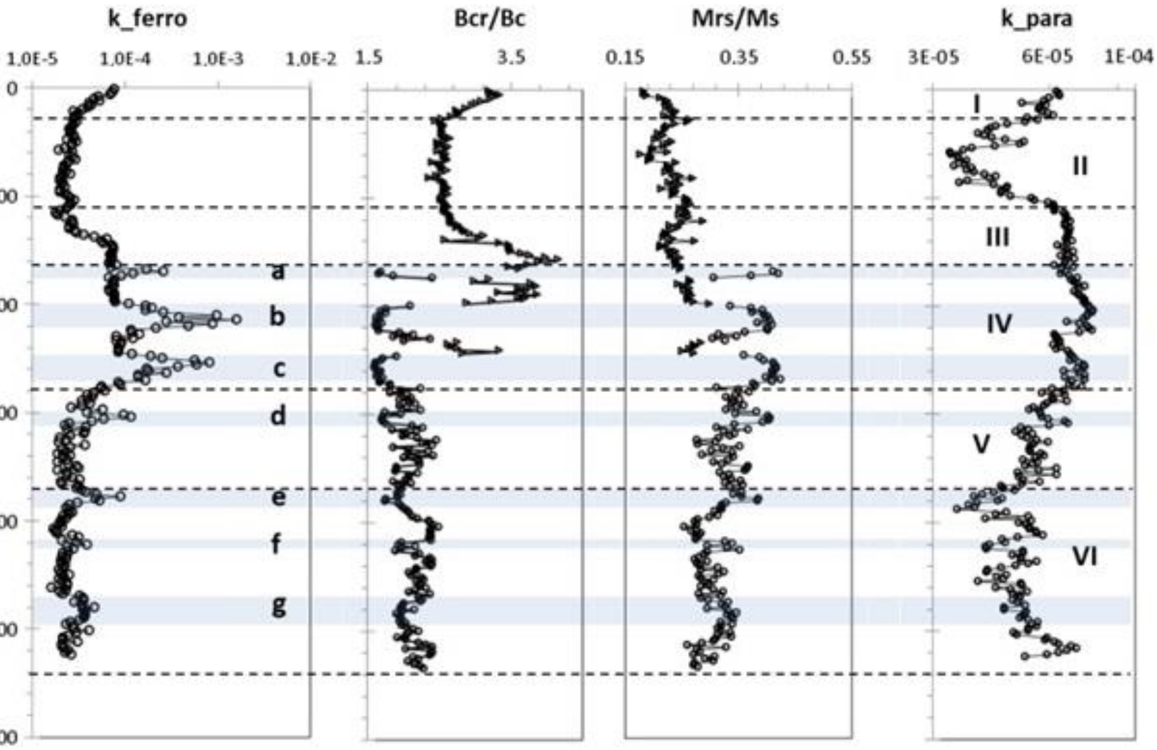


Fig. 8. *Variation of k_ferro and k_para components of MS and Bcr/Bc, Mrs/Ms in core #3. Dashed lines indicate zones (I–IX) identified by changes in paramagnetic susceptibility. The colored areas correspond to zones (a–g), in which SD (biogenic) magnetic grains appear*

Рис. 8. *Вариации ферромагнитной, парамагнитной компонент МВ, Bcr/Bc, Mrs/Ms в керновой колонке #3. Пунктирная линия указывает зоны (I–IX), определенные по изменениям парамагнитной восприимчивости. Цветные области соответствуют зонам (a–g), в которых появляются СД (биогенные) магнитные зерна*

Zone III (114–164 cm) is characterized by steady values of k_para. In this case, a decrease in k_ferro occurs due to a decrease in the number of SP grains. The increase in the content of SP grains in sediments is observed within the interval of (144–164 cm). This may be due to a drop in lake bioproductivity, displacement of OATZ to sediment, and generation of biogenic SP grains.

Zone IV (164–276 cm) is characterized by high values of k_para. Three events leading to an increase in lake bioproductivity (*a*, *b*, *c*) and two events leading to a decrease can be distinguished.

Zone V (276–370 cm) is characterized by an increase in k_para, with a trend towards an increase in the relative content of MD grains.

Zone VI (370–534 cm) is characterized by a decrease in k_para, which is due to a decrease in the total humidity. Moreover, the peaks of the local increase in k_para correlate with an increase in the content of the MD component. This may indicate the final pulses of glacial deposits.

To sum up, such a detailed analysis of lake sediments allowed identification of stages indicating increases and decreases in climate humidity.

Conclusion

MS and hysteresis parameters (M_{rs}/M_s, B_{cr}/B_c) are quite informative and complement each other when being analyzed together in a study of modern lake sediments. Coercive spectrometry data allowed disintegration of the total MS into the paramagnetic and ferromagnetic component, which lead to new information about the nature of environmental changes. The hysteresis parameters (M_{rs}/M_s, B_{cr}/B_c) were used for studying the domain structure of grains and identification of biogenic magnetic grains in the sediment. Six zones were distinguished, differing in humidity and average air temperature, based on variations in the paramagnetic component. Data on the bioproductivity of the lake were derived from SD grain content in the sediment, which also provided information on the nature of climate changes. Joint analysis of paramagnetic and ferromagnetic susceptibility and hysteresis parameters produced information on the effect of total humidity on the bioproductivity of the lake. The next stage of this research will include 1) refinement of the borders separating climate stages based on geochemical studies and 2) correlation with global climate changes based on radiocarbon data.

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Information about the authors

Dilyara M. Kuzina, Cand Sc., Senior Researcher, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. di.kuzina@gmail.com; https://orcid.org/0000-0003-1626-4636

Anastasiia R. Yusupova, Junior Researcher, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. i@ajusupova.ru; https://orcid.org/0000-0001-8732-3053

Nuriia G. Nurgalieva, Dr. Sc., Professor, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. nurgal07@mail.ru; https://orcid.org/0000-0003-4372-9777

Danis K. Nurgaliev, Dr. Sc., Professor, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. danis.nourgaliev@kpfu.ru; https://orcid.org/0000-0003-4269-0962

Pavel S. Krylov, Cand. Sc., Senior Researcher, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. sprintfront@yandex.ru; https://orcid.org/0000-0002-0892-4940

Dinara I. Mulikova, Student, Kazan Federal University, 4/5, Kremlevskaya street, Kazan, 420008, Russian Federation. dindirin8642@gmail.com

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Информация об авторах

Диляра Мтыгулловна Кузина, кандидат геолого-минералогических наук, старший научный сотрудник, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. di.kuzina@gmail.com; https://orcid.otg/0000-0003-1626-4636

Анастасия Рафаилевна Юсупова, младший научный сотрудник, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. i@ajusupova.ru; https://orcid.otg/0000-0001-8732-3053
Нурия Гавазовна Нургалиева, доктор геолого-минералогических наук, доцент, профессор, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. nurgal07@mail.ru; https://orcid.otg/0000-0003-4372-9777
Данис Карлович Нургалиев, доктор геолого-минералогических наук, профессор, директор, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. danis.nourgaliev@kpfu.ru; https://orcid.otg/0000-0003-4269-0962
Павел Сергеевич Крылов, кандидат геолого-минералогических наук, старший научный сотрудник, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. sprintfront@yandex.ru; https://orcid.otg/0000-0002-0892-4940
Муликова Динара Илхомовна, студент, Институт геологии и нефтегазовых технологий, Казанский федеральный университет, Россия, 420008, г. Казань, ул. Кремлевская, 4/5. dindirin8642@gmail.com

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