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MAGNETIC MINERALOGICAL ANALYSIS OF SOILS AS A PART OF THE INTEGRATED GEOLOGICAL AND GEOPHYSICAL MODEL FOR THE HYDROCARBON PROSPECTING

(Представлено членом редакційної колегії д-ром геол. наук, проф. О. М. Карпенком)

Magnetic minerals form or alter in the presence of hydrocarbons, making them a potential magnetic proxy for identifying hydrocarbon migration pathways. We concentrate in the precision magnetic mineralogical analyzes to understand what magnetic minerals in soil from the hydrocarbon fields are responsible for the magnetic signal. To illustrate the results of our studying, we consider the study area of the hydrocarbon deposits near the village Balabanivka, Bogodukhiv district, Kharkiv region. A collection of soil samples is represented by deep medium-humus chernozems. The thermomagnetic analyzes, hysteresis parameters, and isothermal remanent magnetization curves (IRM) were studied for the samples collected near the well (point PR 28) and at the area located out of the hydrocarbon influence zone (point PR 0403). To study the thermomagnetic parameters, different types of the magnetization and magnetic susceptibility (MS, χ), and hysteresis loops we used the Variable Field Translation Balance (VFTB) instrument. The results are confirmed by the highest values of the gas geochemical parameters. The MS variations at temperatures ranging from 200 to 400 ˚C may reflect the presence of either iron sulphides or maghemite. The magnetite occurs for all soils. The wasp-waisted hysteresis loops identified either a mixture of two magnetic minerals (magnetite and hematite) and the admixture of the superparamagnetic and larger grains of the same minerals.

Keywords: tight oil and gas, hydrocarbons, soils, environmental magnetism, magnetic susceptibility, magnetization.

Introduction.

The current situation of the economic development in Ukraine require the development of the oil and gas production. The intensification of the process leads to the need for significant financial costs for energy sector. This situation is considered in the context of national security and requires urgent management decisions, which are based on data from industry organizations and audits. One of the possible ways to increase the production of raw materials of its own origin is not deeply studied sources of compacted oil and gas reservoirs.

Magnetic minerals form or alter in the presence of hydrocarbons, making them a potential magnetic proxy for identifying hydrocarbon migration pathways (*Badejo e al., 2021*). The application of soil magnetism in the oil and gas sector is based on their unique ability to carry information about deep geological processes and near-surface anthropogenic activity (*Menshov, 2018; Menshov et al., 2016*). The physical and chemical basis of the method is the fact of migration of hydrocarbon fluid from the deposit to the upper near surface geological layers as well as to the soil. During the microseepage and the hydrocarbons migration, the fluids generate a change and formation of new magnetic minerals (authigenic minerals). Due to this phenomenon, soils acquire new or altered magnetic properties that can be identified in the field and laboratory conditions. In the process of complex interpretation with other geophysical and geochemical methods, new information on possible deposits of oil and gas is obtained. In addition, information on the magnetic properties of soils allows us to assess the pollution and impact on agronomic properties of lands (*Menshov, 2016*). Such information is especially relevant when perform studies at the areas of non-traditional reservoirs as well as to control the fracturing process.

The fact of uniqueness of the magnetic method is decisive, because the genesis of the deposit does not matter for its application. This means that magnetic studies are independent from the geological nature of the deposit – non-traditional type or classical deposit. Therefore, magnetic methods in the study of areas of compacted oil and gas reservoirs, soils and the upper part of the geological section can use physical and chemical bases and existing developments in relation to classical oil and gas prospecting.

In this paper, we concentrate in the precision magnetic mineralogical analyzes to understand what magnetic minerals in soil from the hydrocarbon fields are responsible for the magnetic signal.

State of the art.

Rock magnetic experiments identify a range of magnetite, maghemite, iron sulphides, siderite, goethite and titanohematite, some of which are part of the background signal, and some due to the presence of hydrocarbons. Typical concentrations of the magnetic minerals are 10–200 ppm. *Badejo e al., 2021* identified an increasing presence of authigenic iron sulphides (likely pyrite and greigite) along the identified lateral hydrocarbon migration pathway. This is likely caused by biodegradation resulting in the precipitation of iron sulphides, however, though less likely, it could alternatively be caused by mature oil generation, which subsequently travelled with the migrating oil to the traps. These observations suggest mineral magnetic techniques could be a rapid alternative method for identifying the severity of biodegradation or oil maturity in core sample, which can then be used to calibrate petroleum systems models.

Kessouri and Ustra, 2019 proposed a laboratory approach that includes quantitative analysis of different magnetic properties: thermomagnetic measurements, frequency dependent susceptibility (FDS) and isothermal remanent magnetization (IRM). We used soil samples from the Brandywine superfund site contaminated with dense non-aqueous phase liquids (DNAPLs) and remediated using amendment injections to enhance the hydrocarbon biodegradation. Thermomagnetic analysis shows a difference between treated and untreated samples: magnetite is observed mostly in the untreated samples, when another magnetic mineral with a Curie temperature around 300°C is observed in the treated samples. This could correspond to a reduced form of magnetite such as pyrrotite or greigite that would indicate microbial reduction.

A combined study of rock-magnetism and electronic-paramagnetic resonance (EPR) was performed by *Costanzo-Álvarez et al., 2019* in core samples from an oil well in the Vaca Muerta Formation (SW Argentina). The aim of this work was to characterize the effects of hydrocarbon-related diagenesis on the magnetic signature of oil shales. The hydrocarbon-induced magnetic anomalies in these oil shales are conditioned mostly by their petrophysical properties. The reactive organic matter free radicals, resulting from the thermal degradation of a kerogen that yields a slightly-biodegraded crude oil, seem to have acted on the primary Fe oxides and sulfides through two diagenetic stages. The second diagenetic stage could have produced partial replacement of framboidal pyrite by authigenic pyrrhotite, as recognized by scanning electron microscopy (SEM), electron X-ray energy dispersion (EDX) experiments and the analysis of the thermomagnetic and IRM curves *De la Rosa et al., 2021* integrate satellite image spectral analyses with rock magnetic and geochemical data (i.e., mass-specific magnetic susceptibility χ, saturation isothermal remanent magnetization SIRM, analysis of SIRM acquisition curves, absorption spectro-radiometry, and X-ray diffraction analyses). This case study illustrates the potential of such an integrated method as an oil exploration tool, and a means to assess the level and scope of the environmental impact produced by hydrocarbon seepage on terrestrial ecosystems.

As was mentioned in introduction, the magnetic studies of the soil at the areas of the compacted reservoirs are important in terms of the environmental issues. *Ayoubi et al., 2020* concluded that through the world as well as in Iran, petroleum hydrocarbons have majority contribution in environmental ricks for human and other organisms. Therefore, they evaluate hydrocarbon pollution effects on soil chemical properties, as well as soil magnetic susceptibility (MS) on a petroleum refinery compound in Isfahan province, Iran. Enhancement of magnetic susceptibility presumably attributed to formation of ferrimagnetic minerals such as magnetite because of degradation of hydrocarbon compounds.

The studies (*Abdelazeem et al., 2021*) aim to detect the Phanerozoic sedimentary sub-basins and their hydrocarbon potentialities within the Qattara Ridge in the northern Western Desert, Egypt, through the interpretation of high-resolution land magnetic data. Reducing gases, coming from the possible underlying hydrocarbon reservoir, play a critical role in the formation of these secondary magnetic minerals. The reservoir in the Qattara Ridge, based on the present study, is characterized by low magnetic susceptibility and low surface magnetic anomaly in the studied locations of productive wells.

Sechman et al., 2020 evaluated the relationship between the distributions of direct and indirect, surface geochemical indices in the selected part of the Outer Carpathians. The research included analysis of molecular composition of soil gas samples and calcite contents as well as the measurements of magnetic susceptibility and pH of soil samples. Comprehensive interpretation of the results obtained by direct and indirect methods in this paper provides another positive example helping to better understand the complicated mechanism of hydrocarbon migration and the potential usefulness of indirect methods suitable for hydrocarbon exploration.

Study Site and Methods.

To illustrate the results of our studying, we consider the study area of the hydrocarbon deposits near the village Balabanivka, Bogodukhiv district, Kharkiv region. A collection of soil samples is represented by deep medium-humus chernozems. The samples were collected within the influence areas of Nedilna-1 (productive) and Nedilna-2 (non-productive) wells.

The geometry of the study site with the sampling points and the map of the magnetic susceptibility is given in Fig. 1 (retrieved from the bachelor diploma of B. Kruhlov). More information about the study site was described in *Menshov, 2021*.

Magnetic mineralogical measurements were performed at the Magnetic Laboratory of the Department of the Earth and Environmental Studies of the Ludwig-Maximilians-Universität München. To study the thermomagnetic parameters, different types of the magnetization and magnetic susceptibility, and hysteresis loops we used the Variable Field Translation Balance (VFTB) instrument. VFTB provide the possibility of the measurements of the magnetization and ma magnetic susceptibility, thermomagnetic curves at-180°C to 800°C (simultaneous measurement of magnetization and susceptibility), isothermal remanent magnetization IRM (full temperature range), backfield (full temperature range), hysteresis loops (full temperature range), and FORC diagrams. VFTB is a sensitive and versatile instrument for measurement of magnetic properties of materials. Due to its high sensitivity, it is an ideal instrument for research in rock magnetism and environmental magnetism and can measure weakly magnetic materials, such as sandstone, limestone, soil etc. It can also be used for magnetic analysis in the field of material science e.g. analysis of synthetic materials, thermal behavior of permanent magnets etc. It is the only instrument on the market that can simultaneously measure the reversible and irreversible components of magnetization and their temperature dependence. This makes it an instrument that is particularly suited for discrimination of different magnetic phases in the sample. RockMag Analyzer software was used to interpret all this data.

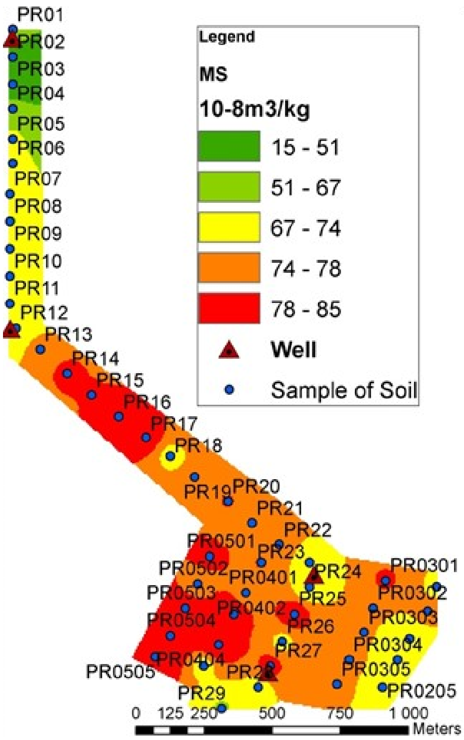

Fig. 1. Study site and the map of thy spatial magnetic susceptibility distribution

Results and Discussion.

The thermomagnetic analyzes, hysteresis parameters, and IRM curves were studied for the samples collected near the well (point PR 28) and at the area located out of the hydrocarbon influence zone (point PR 0403).

We applied the thermomagnetic study of the remanent magnetization to identify magnetic phases, which are related to the natural soil magnetism or induced by the hydrocarbon migration (possibly halo effect). The data processing shows that after heating the sample to a temperature of 700 °C during cooling there is a slight decrease in the magnetization of the samples. There is a decrease in magnetization at a temperature of about 580 °C, which indicates the presence of magnetite as the main magnetic mineral (Fig. 2). The remanent magnetization of the sample collected near well (Fig. 2a) is higher than for the sample collected at the background area (natural soil magnetism, Fig. 2b). The evidence of such kind of the behavior of the remanent magnetization may indicate the direct effect of migrating and microseepage as well as the halo hydrocarbon effect. At the same time at the point PR28 the maxima of gas geochemical parameters were registered.

Magnetite can be formed either by the oxidation of Fe^{2+} or by the regeneration of Fe^{3+} by magnetotactic bacteria (*Lovley et al., 1989*). The magnetotactic bacteria reduce Fe (III) and are able to reduce higher crystalline Fe(III) minerals, such as hematite, goethite and magnetite, as well as poorly crystallized ferrihydrite (*Weber et al., 2006*). From the other site hydrocarbons in sediments and soils also affect Fe(II) oxidants as well as Fe(III) reducing agents. Sequentially, Fe^{2+} can be oxidized by anaerobic and aerobic Fe(II) oxidizing magnetotactic bacteria, which can also direct the formation of magnetite (*Kappler and Straub, 2005*). The mentioned processes can affect the change of ferromagnetic minerals and finally change the magnetic susceptibility of soil under the microssepage.

To go deeper inside the temperature dependence behavior of the magnetic minerals of the soil affected with hydrocarbon migration, we attracted the analyzes of the magnetic susceptibility for the sample PR28. The results are given in Fig. 3.

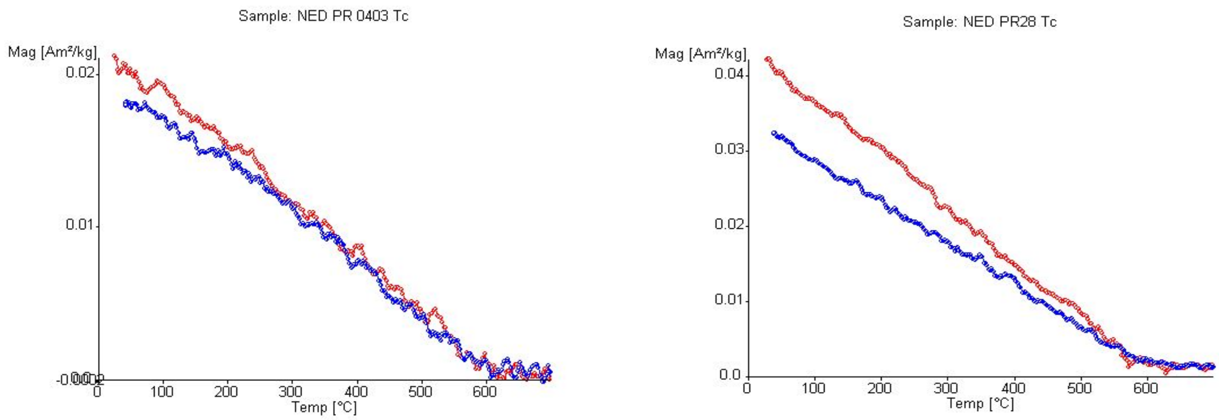

Fig. 2. Temperature dependence of the remanent magnetization:
a – the sample collected near well, b – sample collected at the background area

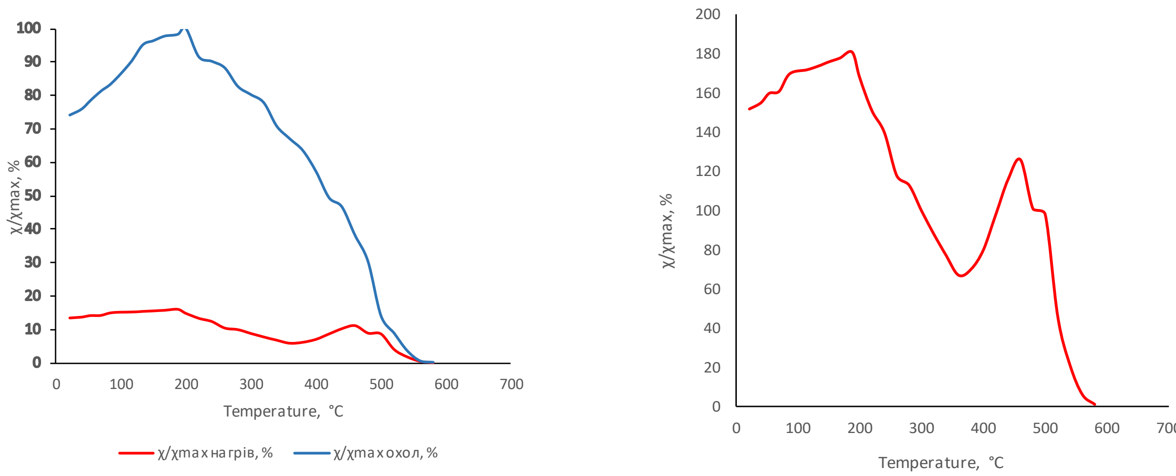

Fig. 3. The emperature dependence of the magnetic susceptibility for the sample collected at the hydrocarbon field near the well, point PR 25:
a – heating and cooling cycle; b – heating in more visible scale

The MS variations at temperatures ranging from 200 to 400 ˚C may reflect the presence of either iron sulphides or maghemite (*Mathé and Lévêque, 2005*). Alternatively, this variation might be due to fine particles staying in a single domain (SD) at room temperature and becoming superparamagnetic at increasing temperatures (*Rijal et al., 2012*). The peak at 500 ˚C could indicate neoformation of magnetite. The latter must have resulted from oxidation of pyrite and other sulphides of hydrocarbon origin transformed into magnetite. The Curie temperature near 580 ˚C suggests the presence of magnetite-like phases. The heating curve (b), however, shows no maximum near 500 ˚C indicating a much smaller content of magnetite. Variation within the temperature range of 200–400 ˚C, on the other hand, is associated with the main magnetic phases – sulphides or maghemites. The first peak at about 200 ˚C could be ascribed to the burning of organic matter, whereas a significant decrease in MS values near 400 ˚C could be related to the effect of maghemite-hematite transformation (*Jordanova and Jordanova, 2016*).

The hysteresis analyzes (Fig. 4) as well as studies of the backfield were applied for the samples PR 0403 and PR28 (see the description above).

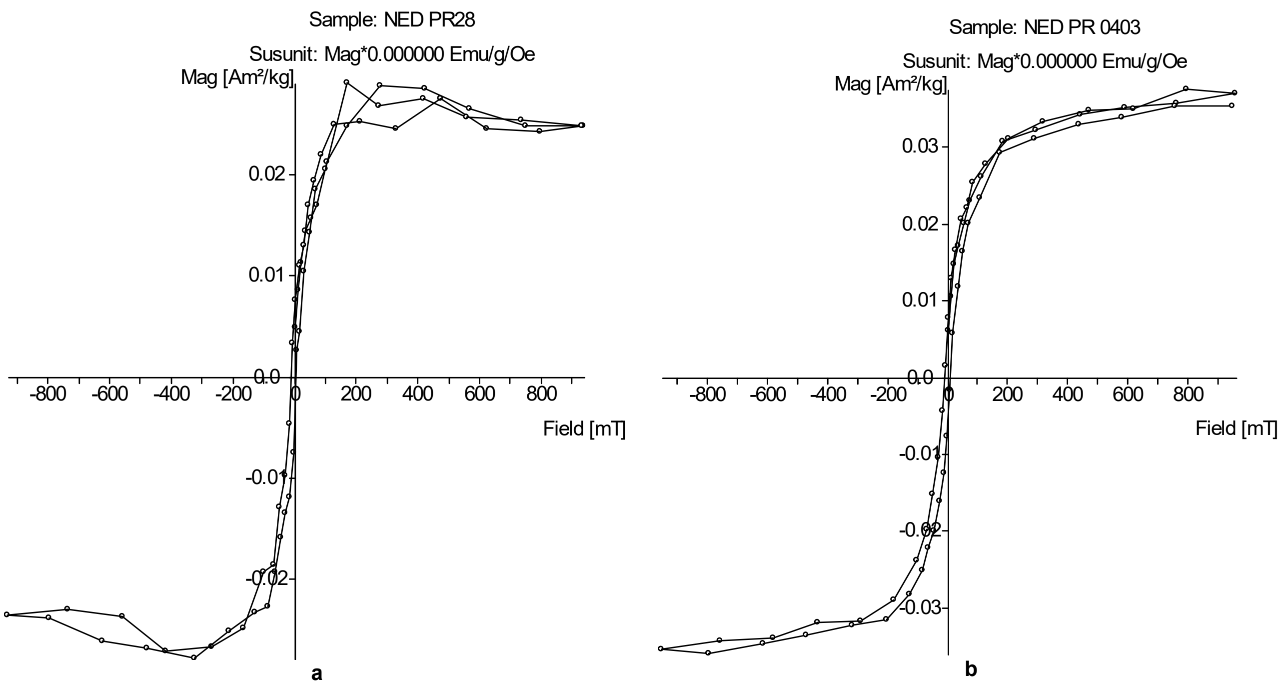

Fig. 4. The hysteresis loops:
a – the sample collected near well, b – sample collected at the background area

The smoothing of the paramagnetic effect was applied. For the sample collected at the point NED PR 0403 the hysteresis loop was obtained at 20 °C. The shape parameter is 0.32. This means the wasp-waisted geometry of the loop. The hysteresis rations are: Mrs/Ms=0.16; Bcr/Bc=2.99; Brh/Bcr=1.86 (where Mrs is remanent magnetization after saturation; Ms is magnetization at the saturation; Bcr is coercivity of remanence; Bc is coercivity; Brh is hard component of the remanent magnetization). The coercivity curve was obtained at 22 °C. Bcr=25.96 mT. According to the mentioned values the magnetics are in the pseudo-single domain state (PSD).

For the sample collected at the point NED PR 28 the hysteresis loop was obtained at 23 °C. The shape parameter is 0.39. This means the wasp-waisted geometry of the loop. The hysteresis rations are: Mrs/Ms=0.20; Bcr/Bc=3.41; Brh/Bcr=0.99. The coercivity curve was obtained at 24 °C. Bcr=24.89 mT. According to the mentioned values the magnetics are in the pseudo-single domain state (PSD).

So-called "wasp-waisted" hysteresis loop is when the central section is smaller than the outer parts. Wasp-waisted loops are typically of mixed ensembles with contrasting coercivities: either a mixture of two magnetic minerals (for example magnetite and hematite) or a mixture of superparamagnetic and larger grains of the same mineral (*Gubbins and Herrero-Bervera, 2007*).

Conclusions.

Magnetic mineralogical studies provide the important data related to the identification of the magnetic phases according to the soil genesis. We studied soils at the areal of the hydrocarbon wells and under the natural pedogenesis. The remanent magnetization of the samples collected near the well was higher than for the samples collected at the background area. In this case the remanent magnetization indicates the direct effect of migrating and microseepage as well as the halo hydrocarbon effect. The results are confirmed by the highest values of the gas geochemical parameters. The MS variations at temperatures ranging from 200 to 400 ˚C may reflect the presence of either iron sulphides or maghemite. The magnetite occurs for all soils. The wasp-waisted hysteresis loops identified either a mixture of two magnetic minerals (magnetite and hematite) and the admixture of the superparamagnetic and larger grains of the same minerals.

The magnetic mineralogical data considered as the complementary method at the complex of the direct hydrocarbon prospecting. Moreover, the natural soil magnetic parameters are important for the proper magnetometry interpretation of the low amplitude anomalies.

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МАГНІТОМІНЕРАЛОГІЧНИЙ АНАЛІЗ ҐРУНТІВ ЯК СКЛАДОВА КОМПЛЕКСНОЇ ГЕОЛОГО-ГЕОФІЗИЧНОЇ МОДЕЛІ ПРИ ПОШУКАХ ВУГЛЕВОДНІВ

Магнітні мінерали утворюються або змінюються у присутності вуглеводнів, тому їх визначення дозволяє ідентифікувати шляхи міграції вуглеводнів. У пропонованій статті наведено високоточний магнітомінералогічний аналіз для розуміння, які магнітні мінерали в ґрунті з родовищ вуглеводнів відповідають за магнітний сигнал. Досліджено ділянку родовищ вуглеводнів поблизу с. Балабанівка Богодухівського району Харківської області. Колекція ґрунтових зразків представлена глибокими середньогумусними чорноземами. Термомагнітні аналізи, параметри петлі гістерезису та криві ізотермічної залишкової намагніченості (IRM) були вивчені для проб, відібраних поблизу свердловини (точка PR 28), та на ділянці, розташованій поза зоною впливу вуглеводнів (точка PR 0403). Для дослідження термомагнітних параметрів, різних типів намагніченості та магнітної сприйнятливості (MS, χ), а також петель гістерезису використовували прилад Variable Field Translation Balance (VFTB). Результати корелюються із високими значеннями геохімічних параметрів газів. Варіації MS при температурах від 200 до 400 °С можуть відображати присутність або сульфідів заліза, або маггеміту. Магнетит зустрічається у всіх ґрунтах. Петлі гістерезису, які нагадують осину стрічку (wasp-waisted), ідентифікували як суміш двох магнітних мінералів (магнетиту та гематиту), а також домішки суперпарамагнітних і крупніших зерен тих самих мінералів.

Ключові слова: ущільнені породи, нафта, газ, вуглеводні, ґрунти, магнетизм навколишнього середовища, магнітна сприйнятливість, намагніченість.