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Spectral Absorption of Unpolarized Light Through Nano-Materials in the Absence of a Magnetic Field

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Abstract:
A study of optical properties, such as light absorption, of a colloidal nano-material, provides information on the biphasic, solid – liquid system microstructure. The nano-material under study is a magnetic liquid (ferrofluid). The disperser agent is petroleum mineral oil and the dispersed material is a brown spar powder (nano-particles). The stabilizer is oleic acid. Light absorption through ferrofluid samples reveals the tendency of solid particles in a colloidal solution to form aggregates. The paper emphasizes the linear dependence between the spectral absorption coefficient, concentration and wavelength. The aggregates cause deviations of the extinction coefficient from values according to the Bouger-Lambert-Beer law. Fe_3O_4 aggregates sized 58.76 nm are formed in the system. The average number of nano-particles forming aggregates is 6. The magnetic liquid to be studied is secure stable and, thus, trustful in technological and biological applications.
Keywords*: Aggregates, Absorption, Biphasic, Colloidal, Extinction.*

1. Introduction

Nano-materials are physical systems whose characteristics are determined by dimensions and structure of nano-particles they include.

The interactions between nano-particles, as well as between nano-particles and disperser liquid agent decide the development process of the system, and its structural transformation into a homogeneous and continuous medium.

In the specific case described bellow, the magnetic liquids (ferrofluids) are biphasic solid – liquid systems, behaving like homogenous mediums, both in the presence and absence of a magnetic field. The present paper observes a system's behavior in the absence of a magnetic field.

The properties of nano-materials depend on the preparation technique (for instance, a sintering procedure), which directly influences the microstructure and the dielectric properties of a nano-material [1]. Properties of some types of nano-material (PbS, PbS/PVA and polyvinyl alcohol) have already been examined [2]. By means of the optical absorption spectrum method, it was determined that the size of nano-particles was 26 Å.

Research regarding absorption of solar radiation through ferrofluids revealed the possibility to use these nano-materials, as thin layers, in thermo-solar conversion [3, 4, 5].

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In biology, nanomaterials are used to select and transport organic cells or viruses. In this case, the dimension of the magnetic nano-particles is within the range of (5 nm to 100 μm). Magnetic liquids suited for biological applications contain nano-particles similar in size with the biological entities: cells (10 – 100 μm, viruses (20 – 450 nm), proteins (5 – 50 nm), and genes (2 nm width and 10 – 100 nm length) [6, 7, 8, 9].

In the present paper, the biphasic systems contain nano-particles of Fe_3O_4, with an average size of 11.354 nm, floating into the carrying liquid, which is petroleum mineral oil. Stabilization of the biphasic system needed fat acid use, namely oleic acid, $C_{17}H_{33}-COOH$. The polar group, $-COOH$, is adsorbed by the brown spar particles. The hydrocarbon string dissolves in oil, insuring the system's stability.

Between the brown spar particles in the ferrofluid London and van der Waals interactions settle. Fig. 1 shows the nature of forces existing inside the ferrofluid, at rest, under a gravitational field: $\boldsymbol{F_1}$ – London force, $\boldsymbol{F_2}$ – van der Waals force, $\boldsymbol{F_3}$ –Archimedes force, $\boldsymbol{F_4}$ – hydrocarbon – hydrocarbon interaction force, $\boldsymbol{P}$ – gravity force. Number 1 denotes magnetic nucleus and number 2 denotes the oleic acid pellicle.

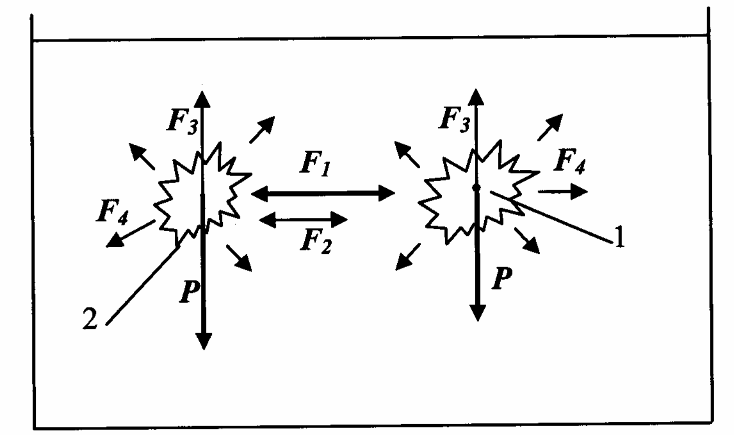

Fig. 1 Forces acting on the brown spar particles in the ferrofluid

As a result of these interactions, especially the London and van der Waals forces, the magnetite particles tend to cluster into aggregates or agglomerates [10]. Single nano-particles, small aggregates with 4-20 nano-particles, and big aggregates with more than 1000 nano-particles co-exist in the colloidal solution [11].

Our previous paper regarding much diluted ferrofluids revealed that the average number of nano-particles in the aggregates is 4 [12].

The structure of magnetic liquids can be studied, by means of different methods, such as optical methods, electronic microscopy and magneto-optical methods. In interaction with light, the magnetic fluid behaves like a continuous medium if the wavelength of the monochromatic radiation is much bigger than the average diameter of the solid particles. So, the magnetic colloid behaves like a homogenous medium and obeys the Bouguer – Lambert – Beer law if the average diameter of the solid particles is less than the tenth part of the radiation wavelength [13, 14].

Previous research revealed that ferrofluid's transparency in a magnetic field depends on magnetic induction [15]. This phenomenon can be used for light signal modulation in the information transmission technique.

The quality of applications involving nano-materials requires preservation of the nano-material, in order to avoid the production of new nano-particle agglomerations. Agglomeration size, established during the elaboration process, must preserve the same value.

Large dimensions of aggregates change the physical properties of the nano-material and make their use difficult for practical purposes.

In the present paper we have examined some optical properties of the magnetic fluid. The work investigates the spectral absorption of unpolarized light in an average concentrated ferrofluid, in the absence of a magnetic field. The entire study is based on the fact that the spectral natural coefficient of extinction for a sample *i*, related to the natural coefficient of extinction for the reference sample, is equal to the unit for all wavelengths and for all concentrations if the dimensions of the nano-particles or aggregates are smaller than the 10^{th} part of the wavelength.

The aim of this work is to calculate the natural coefficient of spectral absorption of the average-concentrated nano-material and to determine the average number of nano-particles from the aggregates that form within this ferrofluid.

It is estimated that in the near future, ferrofluids will be used to develop some new techniques for preparing sintered materials. These sintering technologies will use two properties of ferrofluids [1]:

1. In a non-uniform magnetic field, the apparent density ρ_{ap} of the colloid medium is variable, depending on the gradient of the external magnetic field. If the gradient of the external magnetic field is oriented vertically down, for a non-magnetic particle there are three possibilities: $\rho = \rho_{ap}$ (the particle has a levitation state); $\rho > \rho_{ap}$ (the particle is immersed) and $\rho < \rho_{ap}$ (the particle floats).

2. In a rotational external magnetic field, the ferrofluid also rotates. The angular frequency of ferrofluid is linearly dependent on the frequency multiplied with the intensity of the magnetic field.

1. Sample preparation

The ferrofluid was prepared using the chemical precipitation method. Ferrous and ferric hydroxides result according to the following chemical reaction:

$$FeSO_4 + 2FeCl_3 + 8NH_4OH \rightarrow Fe(OH)_2 + 2Fe(OH)_3 + (NH_4)_2SO_4 + 6NH_4Cl \quad (1)$$

Brown spar, at the temperature of 373 K, is obtained on the basis of the reaction:

$$Fe(OH)_2 + 2Fe(OH)_3 \rightarrow Fe_3O_4 + 4H_2O \quad (2)$$

Brown spar density is higher than petroleum's, so dipol-dipol and van der Waals forces occur between particles. In order to prevent sedimentation and formation of very large aggregats, the brown spar particles are covered with a pellicle of oleic acid, by stirring. The pellicle ($C_{17}H_{33} - COOH$) plays the role of a stabilizer. The polar group $-COOH$ of the oleic acid is adsorbed by the brown spar particles and the hydrocarbon string dissolves in petroleum, to which it is very much alike in composition.

The salts in the solution and porosity in particles were washed away by water at 60 - 80°C. The exceeding oleic acid breaking of brown spar grains needs an acetone addition. The reaction products disperse in oil heated at 120 °C.

Fig. 2 shows the micrography of brown spar nano particles, obtained with an electronic microscope (magnification $6 \cdot 10^4$X). Fig. 2 shows that nano-particles in the ferrofluid obtained using the method described above, are of an irregular shape. For further computations, the brown spar particles are assumed to be spherical. Diameters for 100 nano-particles were measured using optical microscopy and electronic micrographies. The main geometrical feature of nano-particles is proposed to be the average diameter of 100 nano-particles, $\overline{d} = 11.354$ nm [17,18].

The physical characteristics of the magnetic liquid are [16, 17, 18, 19]:

- Saturation magnetization, $\mu \approx 500$ Gs
- Global density, ρ = 1300 kg/m^3
- Concentration of the magnetite nano-particles, c = 550 kg/m^3
- Average dimensions of the brown spar nano-particles, $\overline{d} = 11.354$ nm
- Diffusion coefficient, D = 0.84 m^2/s, at T = 293 K;
- Fluidity, φ = 224.7 ms/kg, T = 293 K;
- Activation energy of diffusion, E_D = 13.57 kJ/mol;
- Activation energy of viscosity, E_η = 13.38 kJ/mol.

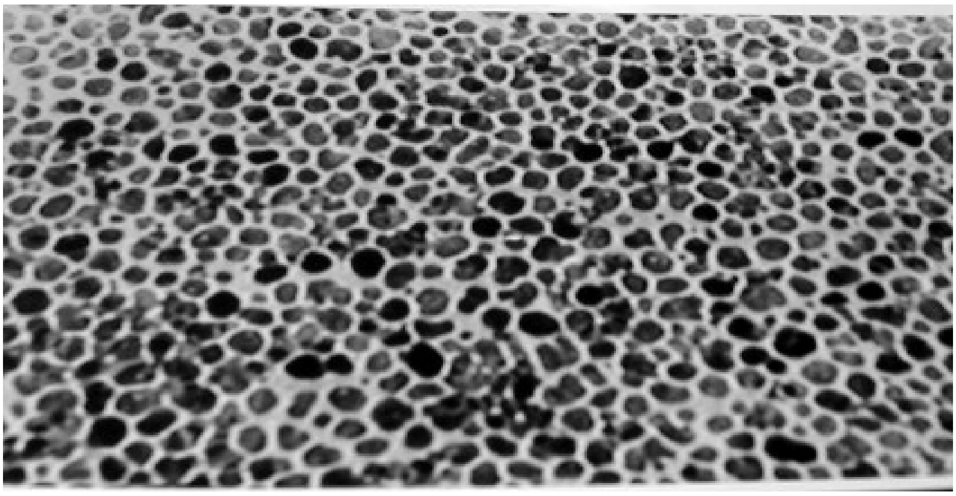

Fig. 2 Micrography of the brown spar nano-particles, achieved with the electronic microscope

Light transmission through ferrofluids is very low. In order to reveal the spectral absorption of light, we diluted the colloidal solution using scattering. Tab. I presents the absolute and relative concentrations of the brown spar in the samples. Sample no.1 was considered to be the reference sample (standard sample).

Tab. I. Concentrations of brown spar in colloidal solutions

Sample's number, i	1	2	3	4	5	6	7	8	9	10	11	12
c_i [kg/m^3]	0.311	0.622	0.933	1.244	1.555	1.866	2.488	3.421	4.043	4.665	5.287	6.220
c_i [r. u.]	1	2	3	4	5	6	8	11	13	15	17	20

2. Experimental Method

The monochromatic light absorption when passing through a homogenous solution is described by the Bouguer-Lambert-Beer law [10, 11, 16]:

$$J_t = J_0 \, e^{-K\,l} \tag{3}$$

where (see, Fig. 3):

J_0 is the intensity of the light passing through the solution

J_t – intensity of the emergent light

l – thickness of the absorbent layer

K – natural absorption coefficient.

The studied liquid is held in a transparent container. The transparent walls absorb and reflect a part of the incident light. In order to eliminate the effect of light absorption and reflection by the container's walls, the light intensities $J_{t,1}$ and $J_{t,2}$ are compared for two containers in which the solution has the same concentration but the thickness is l_1 and l_2. Therefore, the absorption coefficient can be computed using the formula:

$$K = \frac{1}{l_2 - l_1} \ln \frac{J_{t,1}}{J_{t,2}} \ . \tag{4}$$

The absorption coefficient is characteristic to the medium and depends on the light's wavelength. The value of coefficient K is proportional with the solution's concentration c:

$$K = \varepsilon c \ , \tag{5}$$

where ε is the natural coefficient of extinction. It is independent on the concentration, but it depends on the wavelength.

In order to establish if the magnetic liquid behaves like a homogenous solution, the following method was used:

- one of the samples was considered to be the reference sample, (in the present research sample No.1 is the reference sample)
- the extinction coefficient, $\varepsilon_{i,j}$, was explained, with Eqs. (4 and 5), for all concentrations and all wavelengths
- ratios, $Z_{i,j} = \varepsilon_{i,j} / \varepsilon_{r,j}$, were computed for each pair (*i, j*):

$$Z_{i,j} = \frac{\varepsilon_{i,j}}{\varepsilon_{r,j}} = \frac{c_r}{c_i} \cdot \frac{K_{i,j}}{K_{r,j}} \tag{6}$$

The meaning of parameters in Eq. (6) is:

i – index of the sample

j – index of the monochromatic radiation

r – reference sample.

The $Z_{i,j}$ value is the spectral natural coefficient of extinction of the *i* sample, related to the natural coefficient of extinction for the reference sample.

If the solution is homogenous and the aggregate's diameter for the transmitted radiation is smaller than $\lambda_j / 10$, then for the radiation with λ_j as spectral coefficient of extinction, $\varepsilon_{i,j}$ will not change in respect with the variation of the concentration, c_i. For another wavelength, the spectral coefficient of extinction may have another value, $\varepsilon_{i,j} = f(\lambda)$, but the value preserves for all concentrations, $\varepsilon_{i,j} \neq f(c)$. In these conditions, the $Z_{i,j}$ value, computed with Eq. (6), must be equal to unity for a certain fixed value of i and all *j* values.

If the aggregate's diameter exceeds $\lambda_j / 10$, the Bouguer-Lambert-Beer law is not obeyed anymore, and the $Z_{i,j}$ value is too far from the unit. Knowing the minimum wavelength, $\lambda_{\min}$ from which the Bouguer-Lambert-Beer law is obeyed, so that $Z_{i,j}$ moves towards the unit, the maximum diameter of the aggregates can be estimated to $D_{\max} = \lambda_{\min} / 10$. The average approximate number of solid particles, $\overline{n}$, which form an aggregate is $\overline{n} = D_{\max} / \overline{d}$.

The values of $Z_{i,j}$ are dispersed around the value of 1. For $Z_{i,j}$ values, two means can be calculated:

- $\overline{Z}_j$ as the arithmetical mean of $Z_{i,j}$, values (for all concentrations) associated to each wavelength, *j*, of the monochromatic radiation:

$$\overline{Z}_j = \frac{1}{n}\sum_i Z_{i,j}, \quad n = 12. \tag{7}$$

- $\overline{Z}_i$ as the arithmetical mean of $Z_{i,j}$ values (for all wavelengths) associated to each *i* concentration of the biphasic nano-structured system:

$$\overline{Z}_i = \frac{1}{p}\sum_j Z_{i,j}, \quad p = 6. \tag{8}$$

3. Experimental Installation

The absorption of light by the biphasic system was determined with the monochromator PU2. Fig. 3 presents the installation. The components of the installation are: the light source - 1; entrance slit – 2; spherical mirror - 3 (the entrance slit is situated in the focus of the mirror); total reflexion prism – 4 (face AB is silver coated); spherical mirror - 5; exit slit – 6; convergent lens – 7; ferrofluid recipient – 8; photodetector – 9; measuring instrument (microampermeter) -10; micrometric screw for monochromatic wavelength selection – 11 (by means of prism's rotation). A microampermeter indicates the electric intensity of the current generated by the photodetector, *I*. The intensity of the current at the microampermeter is directly proportional with the intensity of the incident light on the sensor, $I \equiv J_t$.

So, equation (4) becomes:

$$K = \frac{1}{l_2 - l_1}\ln\frac{I_1}{I_2} \tag{9}$$

Wavelengths of the selected radiations are presented in Tab. II

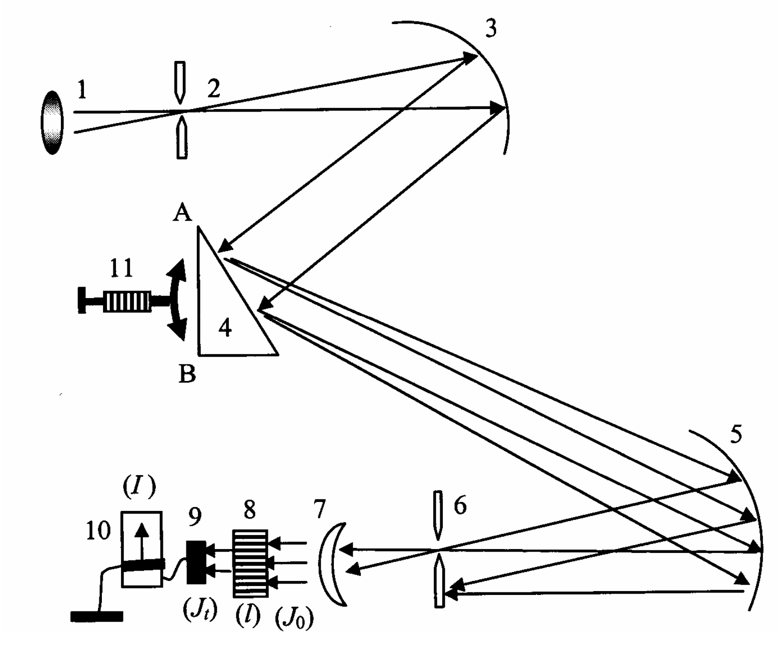

Fig. 3 Installation to measure ferrofluid's transparency

Tab. II. Wavelengths, λ_j.

j [-]	1	2	3	4	5	6
λ_j [nm]	768.2	656.3	587.6	546.1	486.1	434.1

4. Experimental Results

In the visible range, the natural coefficient of absorption for the oil is constant in respect with the radiation's wavelength and has the value $K' = 0.02\,cm^{-1}$ [12].

4.1. Natural coefficient of absorption

The measurements of the absorption of unpolarized monochromatic radiations in the optical spectrum indicated in Tab. II, achieved on the ferrofluid samples indicated in Tab. I were accomplished using the installation presented in Fig. 3. The absorption due to the brown spar particles and agglomerates is equal to the difference between the absorption of the biphasic system and the absorption of the disperser.

Fig. 4 shows the parametric variation of the spectral coefficient of absorption $K_{\lambda,c}$ in relation to the ferrofluid's concentration. The parameter is the monochromatic radiation wavelength.

The wavelengths are given in the box in the left part of Fig. 4. Fig. 4 shows that the natural coefficient of absorption increases in respect with the concentration for all wavelengths. Experimental point groups depicted for each wavelength were processed and trendlines were computed. The slope of lines varies between 0.385 and 0.395 (1/cm). On the other hand, according to equation (5), the slopes of lines represent the mean values of extinction coefficients. For the ferrofluid under study in the paper, the above values correspond to the ones presented in [12,16].

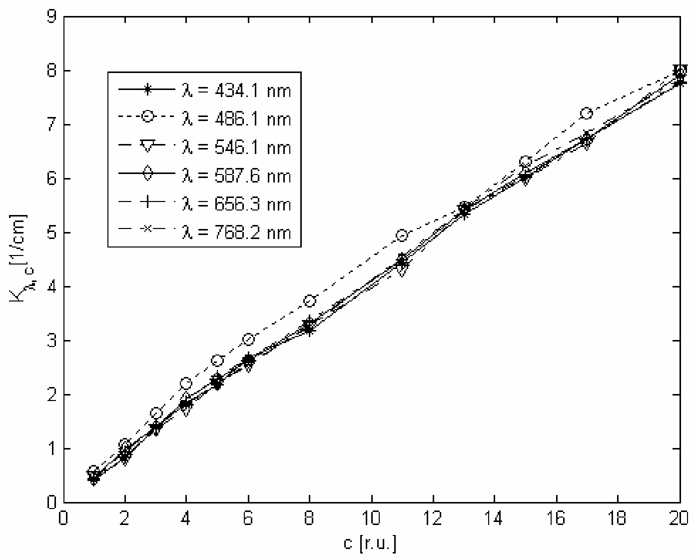

Fig. 4 Dependence of the absorption coefficient in respect to concentration for different wavelengths

Fig. 5 shows the parametric variation of the spectral coefficient of absorption $K_{\lambda,c}$ in respect to the monochromatic radiation wavelength. The parameter is the ferrofluid's concentration.

The concentrations are presented in the box on the right side of Fig. 5. Fig. 5 shows that the natural absorption coefficient slightly increases within the wavelength range of 486.1 – 546.1 nm.

Future developments of this field of research will, therefore, require a careful examination of the conditions that can lead to apparition of a relatively pronounced absorbtion effect in the wavelength range 486.1 – 546.1 nm for high concentrations.

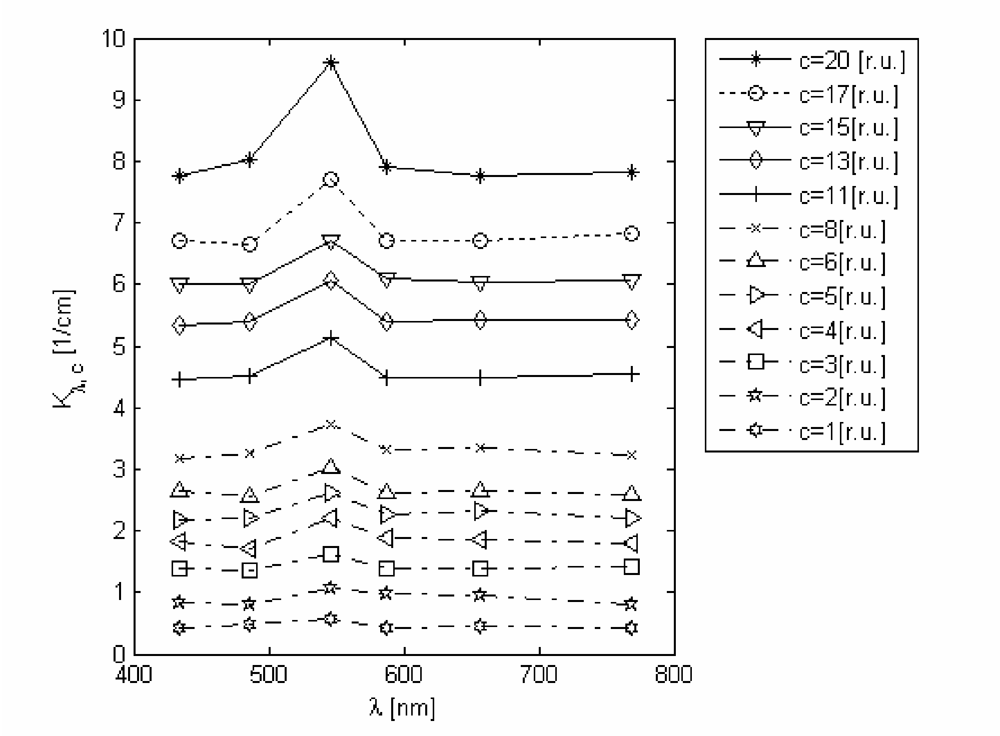

Fig. 5 Dependence of the absorption coefficient in respect to the wavelength for different concentrations

4.2. The values of the $Z_{i,j}, \overline{Z}_j$ and $\overline{Z}_i$ magnitudes

If the solution satisfies the condition $\varepsilon_{i,j} \neq f(c)$, then the $Z_{i,j}$ value must be unitary for all *i* values and for all *j* values. For calculation of the $Z_{i,j}$ value, the sample number *i* = 1 was chosen as the reference sample. Variation of the $Z_{i,j}$ value, in respect to the wavelength for the concentrations mentioned above is presented in fig. 6, $Z_{i,j} = f(\lambda)$. The dispersion of points is bigger for smaller wavelengths and higher concentrations. The points regroup around the unitary value when the wavelength increases and the brown spar concentration in the sample decreases. Maximum deviation from the unit for the points representing the size *Zij* occurs at wavelengths within the range of (486.1-546.1) nm.

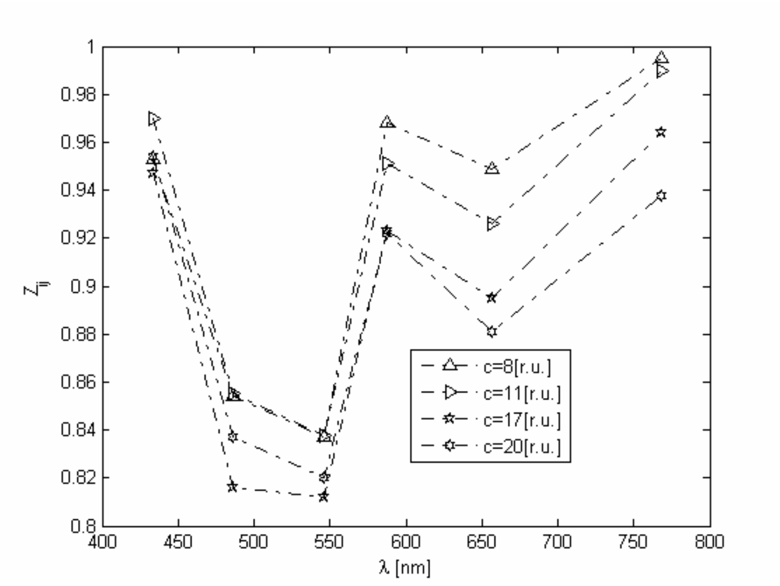

Fig. 6 Variation of $Z_{i,j}$ value in respect to the wavelength for different concentrations

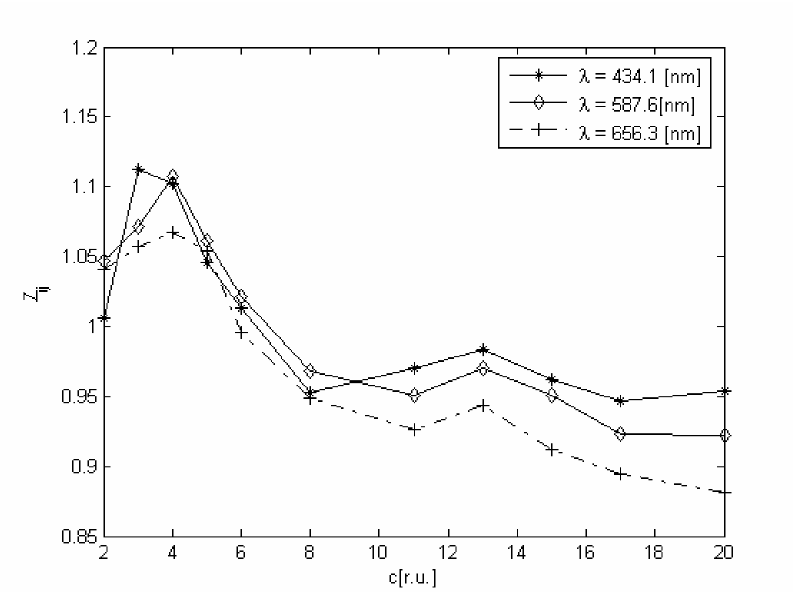

Fig. 7 Variation of $Z_{i,j}$ value in respect to the concentration for different wavelengths

Fig. 7 shows variation of the $Z_{i,j}$ value in respect to the concentration, for different wavelengths, $Z_{i,j} = f(c)$. One can notice the sliding of $Z_{i,j}$ towards smaller values as the concentration increases. As the concentration raises, the point groups are in the region of the value 1, until the concentration reaches 10 r.u. Then, the higher the concentration gets, the more spread the points become. This means that the aggregate dimensions grow proportionally to the colloidal solution concentration.

The dependence of the arithmetical mean values $\overline{Z}_j$ (for all concentrations) in respect to the wavelengths (Fig. 8) shows that we have constant behavior for small wavelengths of the visible spectrum. It also indicates that the dependence $\overline{Z}_j = f(\lambda)$ has an inflexion point in the region of the green spectrum, $\lambda = 587.6\,nm$. For the big wavelengths of the visible spectrum (from yellow to red monochromatic radiation) the value of $\overline{Z}_j$ is around unity, according to the Bouguer-Lambert-Beer law.

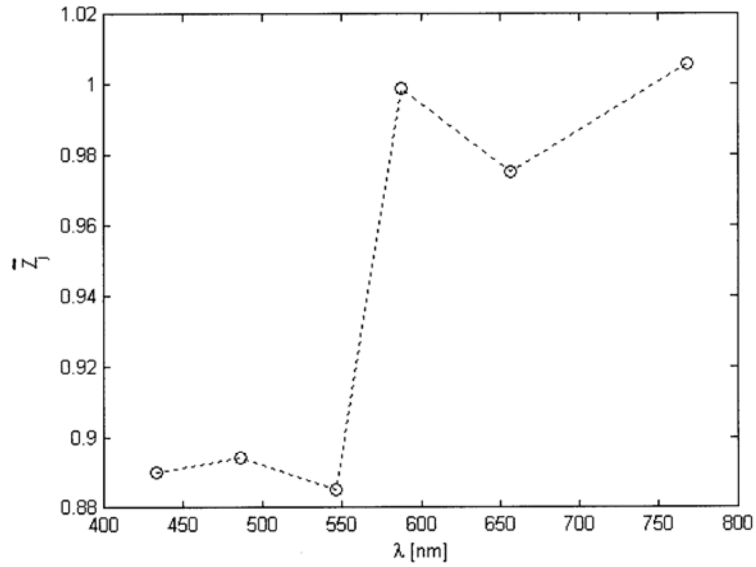

Fig. 8 Variation of $\overline{Z}_j$ value in respect to the radiation wavelength

Fig. 9 shows the variation of $\overline{Z}_i$, in respect to the concentration (for all wavelengths). One can notice that small concentrations are favorable to values close to unity, opposite to the cases when the concentrations are big.

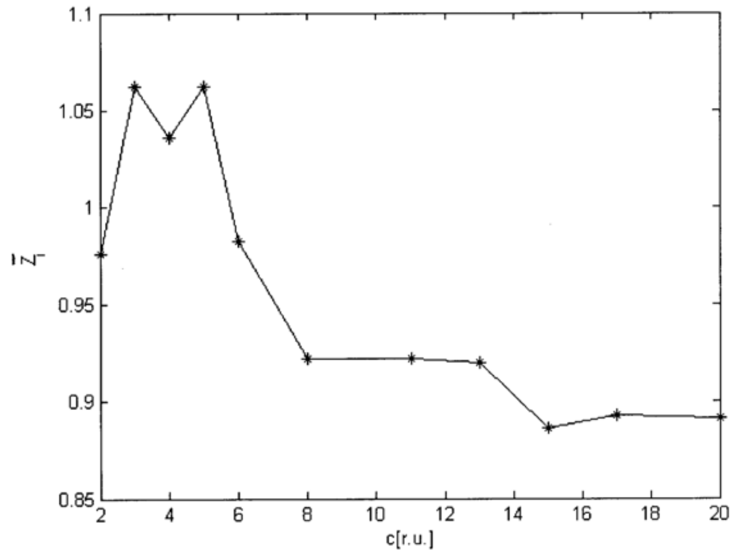

Fig. 9 Variation of $\overline{Z}_i$ value in respect to the concentration of the nano-material

The present paper estimates *λmin* = 587.6 as the minimum wavelength for which the Bouger-Lambert-Beer law is precisely obeyed.

Studies above show that inside the magnetic fluid aggregates form at maximum diameter $D_{\max} = \lambda_{\min}/10$, $D_{\max} = 58.8$ nm. Section 2 demonstrated that the average diameter of a Fe_3O_4 nano-particle, measured by means of electronic micrography is $\overline{d} = 11.354$ nm. The average number of nano-particles in aggregates got in the magnetic fluid, at an average concentration, is $\overline{n} = D_{max} / \overline{d}$, meaning $\overline{n} = 6$. The value of the ratio $\overline{n} = D_{max} / \overline{d}$ was rounded up to the closest entire number.

The paper [18] indicates the minimum diameter (d_1 = 6.2 nm) and the maximum diameter (d_2 = 17.8 nm) of the brown spar particles in the studied forrofluid. Therefore, the maximum number of particles joining to form aggrgates is $n_1 = D_{max} / d_1$, namely, $n_1 = 9$ and $n_2 = D_{max} / d_2$, explicitly n_2 = 4. The relatively small number of Fe_3O_4 particles in the aggregates and the small dimensions of the former, indicate a remarkable stability of the ferrofluid and, thus, a safe use in different technological applications.

6. Conclusion

The concentration of the brown spar particles in the magnetic liquid, in which aggregate formation was studied, varied between 0.311 kg/m^3 and 6.220 kg/m^3. In the magnetic liquid aggregates formed for all concentrations comprised between 0.311 kg/m^3 and 6.220 kg/m^3. The diameter of the aggregates increases as the brown spar concentration grew. The maximum diameter of the aggregates was $D_{max} = 58.8$ nm. The average number of Fe_3O_4 nano-particles gathering into aggregates is $\overline{n} = 6$. The small number of particles in an aggregate indicated good stability of the studied ferrofluid.

The stability of the studied ferrofluid indicates the opportunity of using it in technological applications such as: measurement devices (manometer with variable inductivity), solar collectors, magneto-fluidic valves, magneto-optical transmission of information and others. The magnetic liquid can be used in biological applications such as selection and transportation of proteins, whose dimensions are between 5 and 50 nm.

Further research in this domain should focus on a detailed examination of the ferrofluid's spectral absorption at wavelengths ranging from 486.1 to 546.1 nm. At the same time, future papers should reveal ferrofluid's behavior exposed to light both under a uniform and variable magnetic field.

Once solar energy is implemented, thorough studies get more and more important regarding the behavior of installations using ferrofluid and working in the natural environment.

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Садржај: *Студија оптичких својстава као што је абсорпција светлости колиодног наноматеријала пружа информацију о двофазној микроструктури чврсто-течног система. Проучени материјал је магнетна течност (феро-флуид). Минерално уље је коришћено као дисперзни агент а браон шпар прах (наночестице) је коришћен као дисперзни материјал. Олеинска киселина је коришћена као стабилизатор. Абсорпција светлости кроз узорке феро-флуида је указала на тенденцију формирања агрегата чврстих честица у колоидном раствору. Рад истиче линеарну зависност између спектралног абсорпционог коефицијента, концентрације и таласне дужине. Агрегати узрокују девијацију коефицијента пригушења од вредности у складу са законом Бугерт-Ламбер-Бера. Агрегати* Fe_3O_4 *величине 58.76 нм се формирају у систему. Просечан број наночестица које формирају агрегате је 6. Магнетна течност која се проучана је сигурно стабилна и стога погодна за технолошке и билошке примене.*
Кључне речи*: Агрегати, абсорпција, двофазно, колоидно, пригушење.*