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Modelling optical properties of organic–mineral complexes in water ecosystems

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Abstract
Characteristics of organic–mineral complexes have been theoretically and experimentally studied in the paper, based on the method of integral light scattering indicatrix. Modelling of optical properties was realized using models of the coated spherical particles with a shell which is constant and changeable in the refractive index profile. General regularities have been revealed and generalized parameters for the method of integral light scattering indicatrix have been suggested.

1. Introduction

The problem of determining suspension parameters by light scattering characteristics or the inverse optical problem in turbid media optics relates primarily to the so-called incorrect tasks. It is connected with two main factors. First, natural suspensions are heterogeneous in their composition, forms, sizes, inner particle structure and also in their quantitative composition resulting in a substantial increase in the number of unknown parameters which, as a rule, exceeds the number of constraint equations following from experimental measurements. Moreover, at the same time the problem of solution ambiguity arises, which means that suspensions different in their structure show practically similar optical responses. Second, there are fields of ambiguous solution of an inverse problem, the so-called regions of 'overlapping', even for ideally homogeneous suspensions when the optical response registered within measurement error can correspond, for instance, to different particle sizes. Strictly speaking, just a solution ambiguity conveys the essence of the term 'incorrect problems'.

Historically, the spherical model of suspension particles became the most common one in the dispersion media optics field which was first determined by the rigorous solution of a direct optical problem on the sphere in the year 1908 [1, 2]. However, until the appearance of powerful computers, the calculation of a direct optical problem was a time consuming and complicated process. In the present situation, the approximated calculating methods of the light field scattered by particles, i.e. approximation, has become widespread. It should be noted that just the development of approximation solutions has been allowed to figure directly in the analytical solution of inverse problems. In the rigorous solution, the 'hidden' factors which form the light scattering pattern (size, refractive index) 'come out' in approximations as simple analytical links. The last one particularly allows the correct realization of the experiment in order to avoid ambiguous solutions. In addition, approximations allowed the assessment of the scattered field for particles of nonspherical form that was confirmed both experimentally and by developing rigorous solutions for some nonspherical scatterers, for example, by using T-matrix method [3].

Approximations became especially widespread in the area of optically soft suspensions ($|m - 1| \ll 1$, where $m = m_s/m_0$ is the relative refractive index of particles, m_s is the refractive index of particle matter, m_0 is the refractive index of dispersion medium). Among the well-known approximations, the following should be mentioned: the Rayleigh–Gans–Debye (RGD) approximation, the anomalous diffraction (AD) approximation, the geometric optics approximation and the Wentzel–Kramers–Brillouin (WKB) approximation.

We note a very important result pertaining to the structure formation of the light scattering indicatrix (the angular dependency of the light scattering intensity) and based on approximated solutions. The generalized parameter $u = 2\rho\sin(\theta/2)$ (θ is the scattering angle, $\rho = 2\pi a/\lambda$ is the size parameter, a is the radius of a spherical scatterer, λ is the wavelength of an incident radiation in the medium) plays a governing role in the RGD approximation ($\Delta = 2\rho|m-1| \ll 1$) in the formation of extremes of the indicatrix [1, 4]. Based on the analysis of the WKB approximation which combines AD and RGD [5, 6], in [7] it is shown that with an increase in ρ, this parameter in the small-angle region is reduced to $u = \rho\sin\theta$. So, we can consider that the small-angle structure of the indicatrix is determined by the $\rho\theta$ parameter practically within the entire range of real sizes of natural objects ($\rho \geqslant$ 5–10).

The development of two experimental methods of studying biological disperse systems may be concerned with the main study results of the last ten years which are related to the light scattering indicatrix: the method of integral light scattering indicatrix [8] and the method of the flying light scattering indicatrix (FLSI) [9]. Both these methods are characterized by a continuous angular presentation of the recorded signal. Essentially, the first method uses the link of characteristics of energetic fluxes scattered at different solid angles, with reference to size, structure and type of the matter of suspension particles. The second method is based on the potentials of the flow scanning cytometer and allows for the determination of characteristics of studied particles using calibration coefficients and empirical equations for parameters of differential indicatrix. An exact solution of light scattering on the homogeneous sphere is the theoretical base for both methods and, therefore, the prediction of characteristics of the studied disperse media cannot be completely made by the interpretation of experimental data. Thus, the analysis of structure formation of both differential and integral light scattering indicatrixes of the particles, distinguished from the spherical form or having inner structure, is an important problem.

2. Study objects and methods

Suspended mineral particles which adsorb dissolved organic substances well under natural conditions form complexes—particles of inorganic suspension with a layer of organic substances adsorbed on the surfaces of these particles (organic–mineral detritus (OMD)). As is well known, the boundary surfaces are a concentration of all the interaction processes and promote an accelerated transformation of biogenic matter in the water reservoir [10, 11]. At the same time, matter and energy fluxes which run through these surfaces form a system of boundary layers with abnormal properties and active processes. This results in dividing the aquatic environment into active interaction zones and a comparatively inert inner field in the water reservoir [10]. Therefore, studying boundary surfaces, especially processes of boundary layer formation, their dimensions, structure and composition, and also the biophysical and biochemical processes occurring in them, is an important research task.

Modelling optical OMD properties was realized on the base of two adsorbents (polystyrene latex particles and clay) with humic acid as an adsorbate with concentrations of humic acids in tested samples at 10 and 20 mg l^{-1}. This interval of concentrations of dissolved organic matters is a characteristic of the majority of water reservoirs free from considerable anthropogenic disturbance [12]. Based on assessing adsorbent concentrations made in [13], two concentrations of studied latex and clay particles were chosen: 8×10^9 and 16×10^9 particles/l. The clay fraction had narrow dispersion relatively mean-sized (1.0 μm) particles (the sample is prepared by a deposition method in distilled water; deposition time is from 2 to 6 h). Essentially, this method uses the dependence of deposition rate on particle size resulting in a decrease of recorded suspension turbidity in time, and two latex fractions with mean sizes, d = 1.0 μm (M 150) and 1.5 μm (M 200), where d is the particle diameter with a 6% relative dispersion of sizes.

The models of the coated spheres with shells differing in refractive index profile were used in the theoretical assessment of the light scattering characteristics. Calculation of the algorithm of optical characteristics of the coated concentric particles (where the coated particle is characterized by the following non-dimensional parameters: ρ is the size parameter which corresponds to the outer dimension of particle, q is the ratio of the radii of inner and outer spheres ($0 < q < 1$), $m_1 = (n_1 + i\chi_1)/m_0$, $m_2 = (n_2 + i\chi_2)/m_0$ are, respectively, the complex relative refractive indices of the shell and nucleus matter, m_0 is a refractive index of the medium which we consider a non-absorbing one) was steadily controlled for accuracy using the test system [14]. In all cases, the coincidence of not less than four significant numbers has been obtained.

While calculating coefficients of scattering of a ‘transparent’ spherical particle (the spherical particle with homogeneous nucleus and outer layer, in which the decreasing profile of refractive index heterogeneity was prescribed) the algorithm suggested in [15] was applied for the light scattering of the multilayered sphere. By varying the relative thickness, number of layers and value of the relative refractive index in each layer of such a model, we could obtain different profiles of particle heterogeneity. As a profile of refractive index heterogeneity in the outer layer of the ‘transparent’ spherical particle the profile from [16] was used:

$$m_1(\rho) = \frac{m_2}{1 + b\rho^2}, \qquad b = \frac{m_2 - m_k}{\rho_2^2 m_k},$$

where m_k is a value of refractive index at the boundary of the core and ρ_2 is a size parameter of a particle core.

A steady accuracy control in the calculation process was realized using control tests in the case of the coated sphere [14], as well as for comparing scattering, extinction efficiencies and intensity of light scattering, with literature data available for homogeneous, coated and heterogeneous spheres [14, 16–20]. Assessments have shown that for the given heterogeneity profile it is sufficient to take into consideration 90 layers in order to obtain an accuracy of not less than four significant numbers in all cases.

The main instrumental research method chosen was the integral light scattering indicatrix method. Application of the FLSI method based on the information in the indicatrix extremes is less recommended in the case of particles of irregular form. For example, in figure 1 indicatrixes obtained using the scanning flow cytometer and which are characteristic

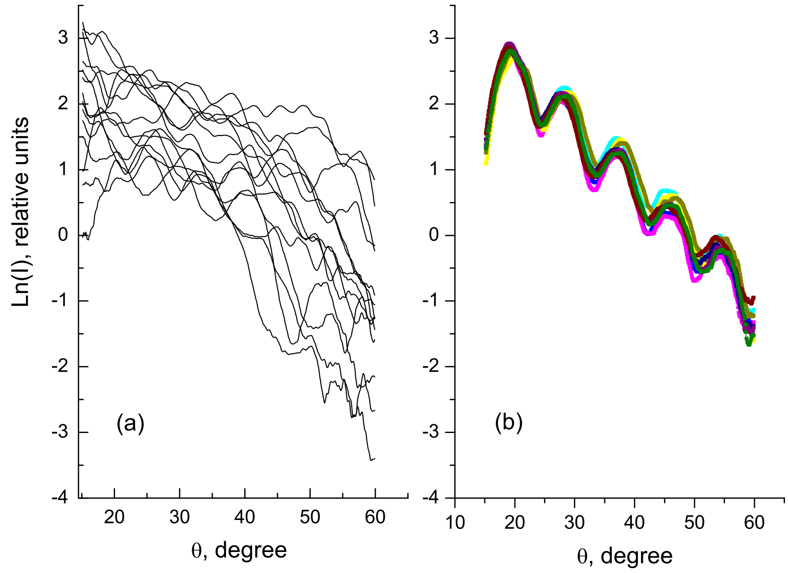

Figure 1. Typical experimental indicatrixes of kaolin particles (*a*) and polystyrene latex particles (*b*) ($m_s = 1.61$) with mean size 2.5 μm (with relative dispersion of sizes 6%) measured by the scanning flow cytometer (wavelength of incident radiation in vacuum $\lambda = 488$ nm). (This figure is in colour only in the electronic version)

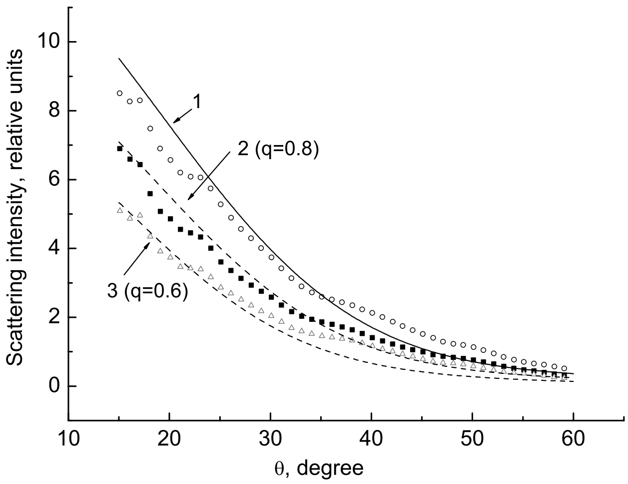

Figure 2. Angular dependences of averaged indicatrixes at different clay (C_c) and humic acid (C_h) concentrations in the studied sample: ○, $C_c = 8 \times 10^9$ particles/l, $C_h = 0$ mg l^{-1}; ■, $C_c = 16 \times 10^9$ particles/l, $C_h = 20$ mg l^{-1}; △, $C_c = 8 \times 10^9$ particles/l, $C_h = 20$ mg l^{-1}. Continuous and dashed curves correspond to theoretical calculations: 1, homogeneous model; 2, 3, the coated model (wavelength of incident radiation in vacuum $\lambda = 488$ nm).

of latex particles (spherical form) and of kaolin particles (irregular form) are presented. It is obvious that in the case of clay particles either data averaging or passing to the more 'stable' integral light scattering characteristics are needed.

Angular dependences of intensity of experimental indicatrixes obtained by averaging intensities of 800 measured indicatrixes for every sample are given in figure 2. The analysis of the presented data shows that the addition of humic acid to the studied samples results in reducing scattering in the range of angles $15^\circ < \theta < 60^\circ$.

Theoretical calculations of scattering indicatrixes for the model of coated and homogeneous spheres with Junge size distribution [12] ($m_2 = 1.15$, $m_1 = 1.05$; $d_1 = 0.5\,\mu$m, $d_2 = 2.0\,\mu$m, where $d_{1,2}$ are minimum and maximum diameters of suspension particles in Junge size distribution) have confirmed the obtained experimental data.

However, the method of integral light scattering indicatrix based on measuring optical response from suspension as a whole, i.e. one primarily averaging the light scattering pattern, can be more efficient in the latter case.

3. Method of integral light scattering indicatrix

Integral light scattering indicatrix $F(\theta_0)$ presents a flux share which is scattered to the cone with the cone angle $2\theta_0$ to the whole flux of the scattered energy:

$$F(\theta_0) = \frac{2\pi \int_0^{\theta_0} I(\theta)\sin\theta\,\mathrm{d}\theta}{2\pi \int_0^{\pi} I(\theta)\sin\theta\,\mathrm{d}\theta},$$

where $I(\theta)$ is scattering intensity in the direction θ.

In [21] it is shown that in RGD approximation, the integral light scattering indicatrix of the homogeneous sphere is invariant in the coordinates $\rho\theta_0$ and is described by the expression ($\theta_0 \ll 1$):

$$F\left(2\rho\sin\frac{\theta_0}{2}\right) \approx F(\rho\theta_0) = \frac{2}{9}\left\{(\rho\theta_0)^2 - \frac{1}{10}(\rho\theta_0)^4 + \cdots + O((\rho\theta_0)^4)\right\}. \quad (1)$$

Equation (1) is valid with an error less than 10% for $\rho \geqslant 5$; with that the error decreases with increasing ρ. Moreover, adaptability of the equation (1) for describing the integral light scattering indicatrix goes beyond the limits of RGD theory adaptability ($\Delta \ll 1$) and increases up to the value $\Delta \leqslant 3$–4.

Based on the behaviour of the integral light scattering indicatrix, the theoretical method of determining mean particle size by the half level of scattering which is observed at $\rho\theta_0 = 1.75$ has been elaborated in [21].

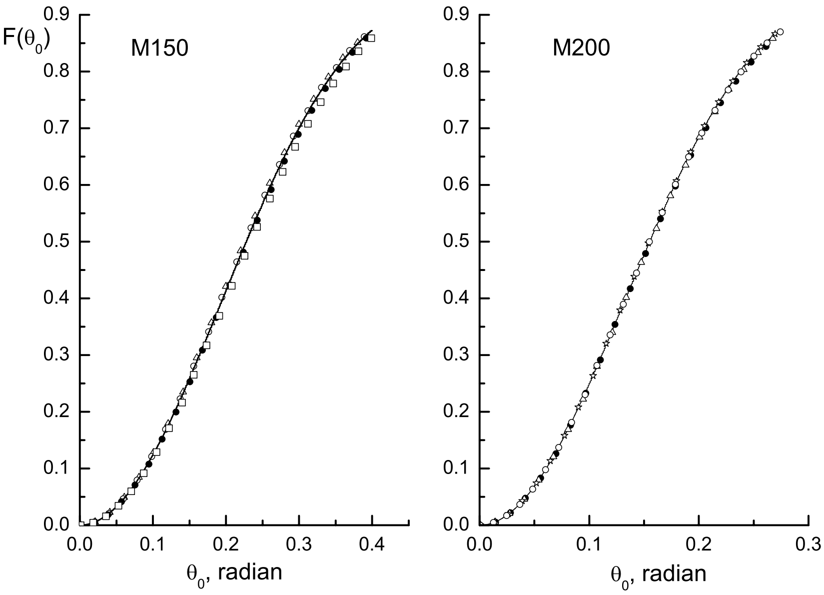

Figure 3. $F(\theta_0)$ for different concentrations of humic acid (C_h) and two types of latex particles with two concentrations (C_l) in the studied sample (wavelength of incident radiation in vacuum $\lambda = 550$ nm). ——, $C_l = 8 \times 10^9$ particles/l, $C_h = 0$ mg l^{-1}; ●, $C_l = 8 \times 10^9$ particles/l, $C_h = 10$ mg l^{-1}; △, $C_l = 8 \times 10^9$ particles/l, $C_h = 20$ mg l^{-1}; □, $C_l = 16 \times 10^9$ particles/l, $C_h = 10$ mg l^{-1}; ○, $C_l = 16 \times 10^9$ particles/l, $C_h = 20$ mg l^{-1}.

Table 1. Mean size d_{av} obtained using the method of integral light scattering indicatrix at different latex and humic acid concentrations in the studied sample.

C_l (particles/l)	C_h (mg l^{-1})	d_{av} (μm) M 150	d_{av} (μm) M 200
8×10^9	0	1.015 ± 0.070	1.441 ± 0.020
8×10^9	10	0.992 ± 0.030	1.431 ± 0.060
8×10^9	20	1.032 ± 0.020	1.416 ± 0.080
16×10^9	10	0.977 ± 0.050	1.443 ± 0.040
16×10^9	20	0.995 ± 0.070	1.447 ± 0.050

3.1. Latexes

Latex particles are used as calibration material for analysing different microparticles and as convenient objects for research. The theoretically calculated dependences of the light flux $F(\theta_0)$ for two types of latex particles with sizes obtained using the method of the integral indicatrix at different latex and humic acid concentrations in the studied sample are given in figure 3 and table 1. The analysis of presented information shows that the modifications of the θ_0 angle value for all the varied concentrations of latex and humic acid in the studied sample do not exceed 3.5% which corresponds to a permissible error of the integral indicatrix method. From here it follows that latex particles have a low sorption ability (a very slight layer of adsorbed matter is formed on their surface), and it is not possible to catch such small changes in the dimension of the studied particles using the method of integral indicatrix. However, the last problem may be successfully solved using the FLSI method [22].

3.2. Clay

In contrast to latex particles, kaolin suspensions possess a large adsorption ability. Spectra of the absorption of humic acid solution with added clay suspension before and after filtration are given in figure 4. Because of the decrease of adsorption efficiency after filtration and also the concentration of dissolved organic matters, we can conclude that the humic acids have adsorbed on the suspension particles.

Let us assess theoretically the possibility of using the method of the integral light scattering indicatrix in order to determine OMD particle size, i.e. an error in determining $\rho\theta_0$ which corresponds to the half level of $F(\theta_0)$. Let us take a homogeneous spherical particle as a model, with the refractive index averaged over the volume of the particle, also the coated concentric and 'transparent' spherical particles.

The mineral particle which has adsorbed organic matter can be presented in the first approximation as a model of the coated particle with a fixed nucleus size ($d \approx 1\,\mu$m, $m_2 = 1.15$) and with varying thickness of the outer shell. We assume that

$$\alpha_1 = \frac{V_1(m_1 - 1)}{2\pi} = \text{const}, \tag{2}$$

where V_1 is the volume of the shell, m_1 is a relative refractive index of the shell and α_1 is its volume polarizability [1]. This condition corresponds to the following physical picture: with increasing shell thickness, the tenuity of adsorbed organic matter also increases (but the amount of adsorbed matter is constant). Let us take as an example, the instance when the maximum value of the complex relative refractive index in the outer layer is $m_{1\max} = 1.05 + \mathrm{i} \cdot 0.01$ ($q = 0.9$). As seen from data presented in figure 5(*a*), at increasing shell thickness, the error in determining $\rho\theta_0$ of the coated model with regard to the homogeneous one with the refractive index averaged over volume also increases.

From the physical essence of the principle of the RGD approximation, we should expect that the behaviour of the spherical particle with the refractive index which varies freely

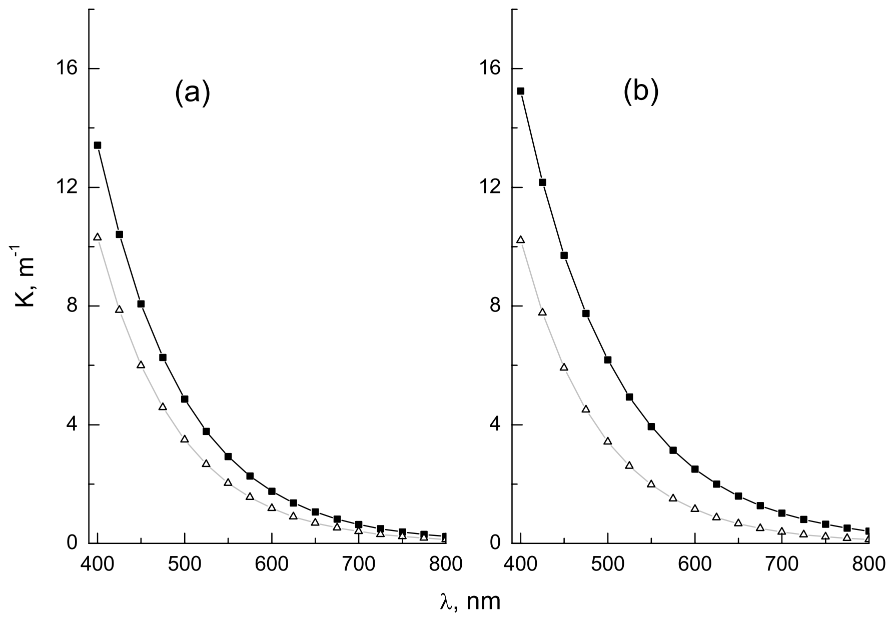

Figure 4. Spectra of absorption (K) with different concentrations of humic acids and clay at filtering: (a) ■, $C_h = 10\,\mathrm{mg\,l^{-1}}$, $C_c = 5.9 \times 10^9$ particles/l; △, filtrate of primary solution through the filter 0.4 μm; (b) ■, $C_h = 20\,\mathrm{mg\,l^{-1}}$, $C_c = 5.9 \times 10^9$ particles/l; △, filtrate of primary solution through the filter 0.4 μm.

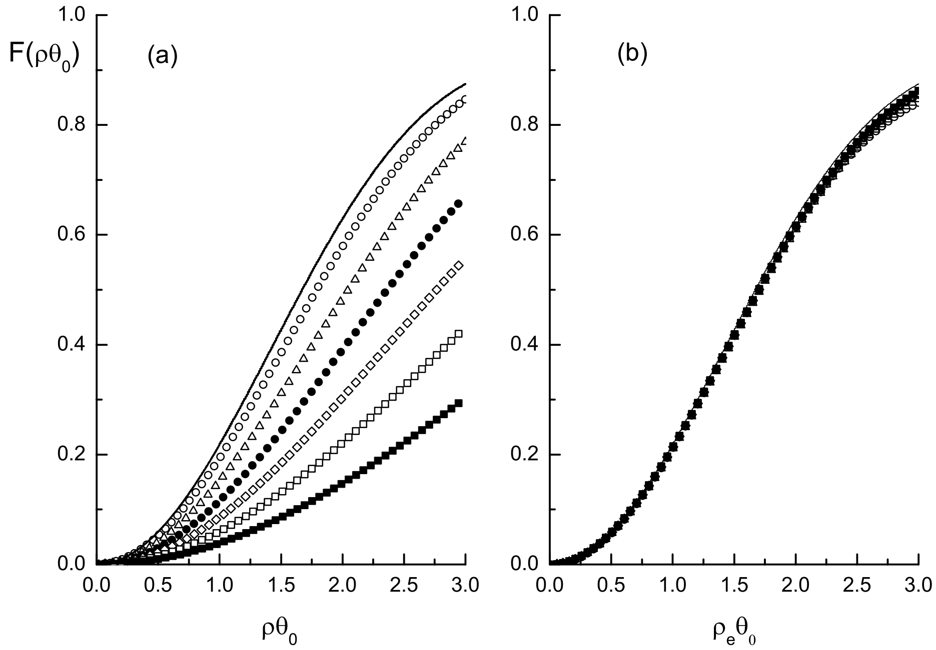

Figure 5. Integral light scattering indicatrix calculated for: (a) homogeneous spherical particle with m averaged over particle volume (——) and for the coated spherical particle with different outer layer width (○, $q = 0.9$; △, $q = 0.8$; ●, $q = 0.7$; ◇, $q = 0.6$; □, $q = 0.5$; ■, $q = 0.4$) in coordinates $\rho\theta_0$; (b) the same in coordinates $\rho_e\theta_0$. $\rho_2 = 7.62$, $m_2 = 1.15$, m_1 is calculated from condition (2).

radially will be identical to that of the homogeneous sphere, the only difference being that, instead of the generalized parameter $\rho\theta_0$, we need to use $\rho_e\theta_0$.

The search for ρ_e may be performed based on the following assumptions. As is well known, for homogeneous spherical particles, up to 90% of scattered energy is concentrated in angles upto the first minimum. Equating the angle positions at which the first minimum appears in the scattering intensity of homogeneous and coated spheres, and later knowing that for the homogeneous spherical particle, the first minimum appears at $\rho\theta \approx 4.49$ in the RGD domain, enables us to find ρ_e. The comparison of the integral light scattering indicatrix of a homogeneous sphere, with refractive index averaged over volume, and that of the coated sphere which was calculated in effective coordinates $\rho_e\theta_0$ is shown in figure 5(b). It is obvious that the integral indicatrix for the coated spherical particle in the pointed coordinates practically coincides with $F(\rho\theta_0)$ of the homogeneous sphere with refractive index averaged over

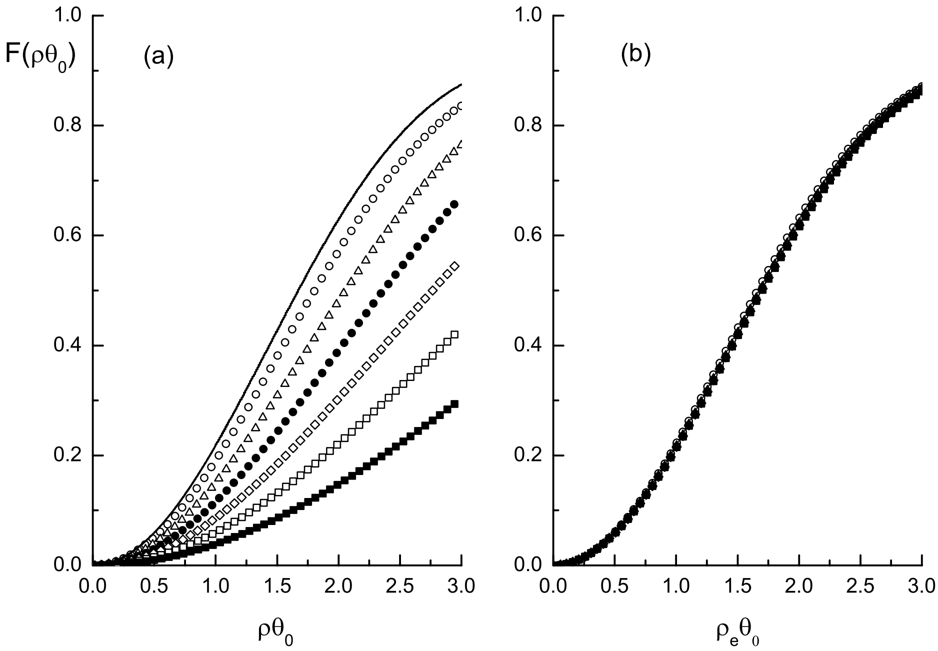

Figure 6. Integral light scattering indicatrix calculated for: (*a*) homogeneous spherical particle with m averaged over particle volume (——) and for the 'transparent' spherical particle with different outer layer width (○, $q = 0.9$; △, $q = 0.8$; ●, $q = 0.7$; ◇, $q = 0.6$; □, $q = 0.5$; ■, $q = 0.4$) in coordinates $\rho\theta_0$; (*b*) the same in coordinates $\rho_e\theta_0$. $\rho_2 = 7.62$, $m_2 = 1.15$, m_1 is calculated from equation (3).

its volume. Thus, the theoretical evaluations have shown that volume which corresponds to ρ_e contains up to 90% of the entire scattering substance.

Using the model of the 'transparent' spherical particle and assuming that

$$\alpha_1 = \frac{1}{2\pi}\int_{V_1}(m_1 - 1)\mathrm{d}V = \text{const}, \qquad (3)$$

the data presented in figure 6 (at $m_k = 1.05 + \mathrm{i}\cdot 0.01$) are obtained.

As we can see from the data presented in figure 6(*a*), at increasing shell thickness the error in determining $\rho\theta_0$ of the 'transparent' model also increases with regard to the homogeneous one with a refractive index averaged over particle volume. Since the angular dependence of scattering intensity of the 'transparent' particle can have 'smoothed' extremes, we shall choose ρ_e such that the truncated model contains 90% of the entire scattering substance. In figure 6(*b*), the integral light scattering indicatrixes of the homogeneous particle, with refractive index averaged over its volume, and the 'transparent' particle are presented, assuming that $F(\rho\theta_0)$ of the 'transparent' particle was calculated in effective coordinates $\rho_e\theta_0$. In recorded coordinates, the $F(\rho\theta_0)$ of the 'transparent' particle coincides with the integral indicatrix of the homogeneous particle.

Thus, the above given theoretical analysis has shown that in effective coordinates $\rho_e\theta_0$, the integral indicatrix of the structured particle (two-layered, 'transparent' particles) with only small error corresponds to $F(\rho\theta_0)$ of the homogeneous particle. Using table 2, it is possible to assess the error in the determination of the mean effective size by the method of integral indicatrix which arises when the inner structure of suspension particles is not taken into account.

Table 2. Comparison of ρ and ρ_e calculated theoretically for the model coated and 'transparent' particles.

	Coated particle		'Transparent' particle	
q	ρ	ρ_e	ρ	ρ_e
0.9	8.47	7.93	8.47	7.88
0.8	9.53	7.95	9.53	7.90
0.7	10.89	7.97	10.89	7.94
0.6	12.70	7.99	12.70	7.95
0.5	15.24	8.00	15.24	7.96
0.4	19.05	8.03	19.05	7.99

In table 2, ρ values and appropriate ρ_e values at various q are presented for models of the coated and 'transparent' particles. As can be seen, the nucleus size is determined with a mean error of 5%. This can be explained by the fact that the volume polarizability of the outer layer at conditions (2) and (3) does not largely contribute to the total volume polarizability of the particle.

The fact that the particle size recorded in the method of integral light scattering indicatrix corresponds to the radius, within which practically all the scattering substance lies, results in the following assessment of ρ_e:

$$\rho_e = \frac{\rho q\cdot\alpha_2 + \rho\cdot\alpha_1}{\alpha_2 + \alpha_1}, \qquad (4)$$

where $\alpha_2 = V_2(m_2 - 1)/2\pi$ is the volume polarizability of the nucleus, and $\alpha_1 + \alpha_2$ is the volume polarizability of the whole particle. In other words, ρ_e is obtained from the principle of averaging in particle polarizability.

At the dominating nucleus, volume polarizability ρ_e will be determined with a small error by nucleus size but at the dominating shell volume polarizability, $\rho_e \approx \rho$. In

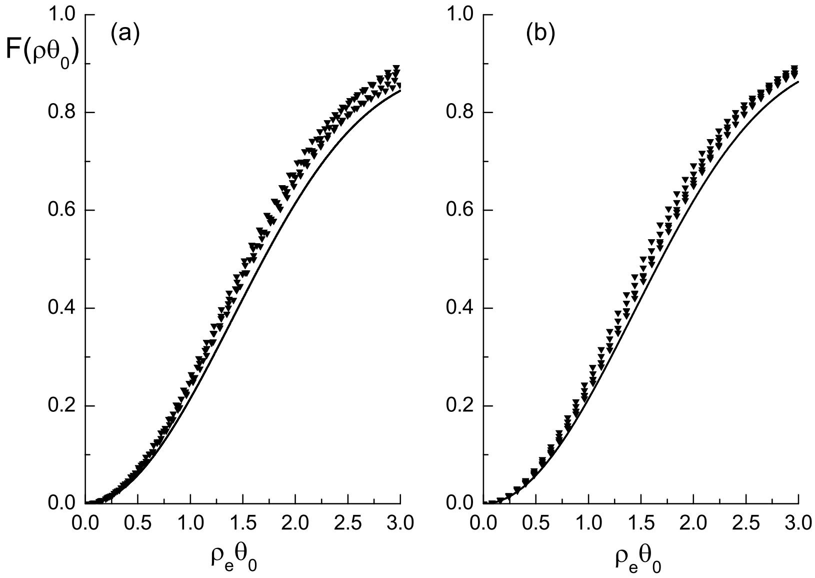

Figure 7. Theoretically calculated integral light scattering indicatrix for homogeneous (——) and the coated spherical particles (▼) for different values of q, refractive indices of nucleus and shell. (*a*) The shell volume polarizability is 3 times more than the nucleus volume polarizability; (*b*) the shell volume polarizability is 2 times more than the nucleus volume polarizability.

intermediate cases, however, the ρ_e value depends on the values of ρ and ρq in (4).

Presented in figure 7 are the theoretically calculated $F(\rho\theta_0)$ for the homogeneous and coated concentric particles for the cases when the ratio of the volume polarizability of the outer layer to the nucleus equals 2 and 3, respectively, at different values of q and refractive indices of nucleus and shell. The $F(\rho\theta_0)$ of the coated particle is given in effective coordinates $\rho_e\theta_0$, where ρ_e is calculated using (4). The analysis of the presented data shows that the relative error of θ determination, which corresponds to the half level of $F(\rho\theta_0)$, does not exceed 10% for the coated particle. It is obvious that with further increasing contribution of the shell volume, the polarizability ρ_e will approach ρ according to (4).

In real natural systems and model experiments the refractive index of organic matter adsorbed on suspended mineral particles is comparable with the refractive index of suspension particles, owing to the high concentration of organic matter on the surface of suspended particles as compared with its concentration in the solution. Hence, using the method of integral light scattering indicatrix for determining OMD dimension with a small error, the whole size of a complex particle is being obtained.

4. Conclusion

At present, a rich experimental material has been accumulated in the field of optics of turbid media. New original devices for optical measurements have been constructed. However, it should be noted that, on the whole, the experimental base is much ahead of the theoretical one owing either to the absence of a precise theory of light scattering for arbitrary particles or to objective difficulties of inversing optical information in cases where there is such a decision to be made.

Some ideas for promising methods based on measuring light scattering indicatrixes have been outlined in this paper. The universal coordinates for representation of F have been shown. Using approximated methods for assessing the scattered field, these ideas not only allow solving inverse problems rather efficiently but also promote the understanding of mechanisms which govern the processes of formation of optical responses. It is obvious that the combination of approximated approaches and precise theory permits the solving of inverse problems and the realizing of experiments more effectively.

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