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ANALYSIS OF THE SOLUTION OF THE ELASTIC LIGHT SCATTERING INVERSE PROBLEM FOR POLYMERIC EMULSIONS

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ABSTRACT

We consider the problem of inverting elastic light scattering (ELS) measurements from polymeric emulsions, to obtain its particle size distribution (PSD) and its refractive index. The mathematical formulation results in a nonlinear inverse problem. A Fredholm integral equation of the first kind appears with an unknown parameter in its kernel.

We discuss the existence, uniqueness and stability of the generalized solutions that can be obtained when the problem is stated as a minimization problem with a least square functional.

First, we assume that the distribution is known, and for this case we prove that the solution exists and is unique as long as the relation between the measurements and the parameter is by an injective function. Then, we use this result to state sufficient conditions for the complete problem. The analysis of existence and uniqueness of the solution for the problem in hand is supported by numerical simulation.

The Phillips-Tikhonov regularization form is proposed to stabilize the problem when noisy-data is available.

NOMENCLATURE

a	particle ratio
D	particle diameter
f	particle size distribution (PSD)
g	noisy ELS measurement
I	light scattering intensity
J	functional
L	smoothing restriction on f
m	relative particle refractive index
n_p	particle refractive index
n_s	solvent refractive index
S	Lorentz-Mie scattering function
T	Operator that represents the integral equation

Greek Letteres

ε	experimental error
γ	regularization parameter
λ	wavelength of the incident beam
θ	scattering angle

Superscript

*	adjoint
T	transposed

INTRODUCTION

The Light Scattering is a well-established technique for characterizing micro particles due to its sensitivity to size, structure, shape and orientation. The method, moreover allows measurements to be carried out rapidly, nondestructively on remote or in situ specimens. As a consequence light scattering instruments are widely used in physics, environmental science and engineering. The technique nevertheless suffers from a major disadvantage, which is that no immediate inversion procedure exists by which the properties of the particle –its radius and refractive index- can be obtained from an experimental scattering pattern. This applies even when the specimen geometry is one for which a rigorous treatment of light scattering is known, that is, the isolated homogeneous and isotropic sphere. The theory for this case was given in Mie's paper [1], but it did not include the study of the inverse problem.

Many articles have reported during the last two decades various heuristic and quasi-analytic strategies to solve the problem of inverting light scattering measurements. The earliest attempts were based on the assumption that the refractive index of the particles was known [2-4]. More Recently, Ludlow and Everitt (1900)[5] considered the case of a homogeneous sphere and derived a mathematical construction to invert the Mie coefficients to find the refractive index and the radius of the particle. The same problem was treated numerically by Zakovic et al. (1898) [6] and by Hodgson(1900) [7] .

We are interested in the problem of inverting the light scattered by a collection of spherical homogeneous particles of different sizes suspended in a medium. Particulate materials, such as powders, sprays, emulsions, suspensions and solutions occur in polymer science. They are characterized by the particle size distribution (PSD). Different light scattering techniques exist for sizing this type of materials. We consider elastic light scattering (ELS) that covers a diameter range from about 100 nm up to a few micrometers [8]; many polymeric emulsions have particles with diameters inside this range.

The problem of obtaining the PSD and the refractive index for this kind of materials was solved through different comprehensive methodologies in some previous articles ([9],[10]). More recently [11], we proposed an alternative procedure and presented the solutions found for some particular polymeric emulsions .

The purpose of this paper is to discuss existence, uniqueness and stability of the solutions obtained when the inversion problem is formulated as an optimization problem, considering ideal measurements, as well as perturbed ones.

MATHEMATICAL DESCRIPTION OF ELASTIC LIGHT SCATTERING MEASUREMENTS

As an electromagnetic wave propagates through a medium, the wave is attenuated. The attenuation of electromagnetic energy is known as extinction and is the result of two different mechanisms: absorption and scattering. Energy that is absorbed is converted into some other form such as thermal or chemical energy, while the energy that is scattered is merely redirected and remains in electromagnetic form. The frequency of the scattered electromagnetic wave is generally the same as the frequency of the incident wave. This is referred to as elastic scattering.

Consider a beam of light incident on an arbitrary particle. The superposition of the incident light and of all the secondary reradiated waves gives the total scattered light. The scattered light varies with the scattering direction, with the size, shape, orientation, and optical properties of the particle and with the frequency, irradiance and polarization of the incident beam. The problem is too complex unless restrictive assumptions are made. This work relies on the following assumptions. First, only elastic light scattering is considered. Second, if the scattering is the result of more than one particle, then the scattering is independent, i. e. the effect of the various particles may be added, and the multiple scattering effect is neglected. Third, the incident beam is monochromatic. These assumptions reduce the light scattering problem to that of finding the electromagnetic field inside a particle and in the medium surrounding the particle. Due to the mathematical complexity, analytical solutions to the light scattering problem have only been obtained for particles with simple geometries and properties.

Mie theory provides the most important of the analytical solutions; it describes the electromagnetic field scattered by a homogeneous, isotropic sphere of arbitrary radius, *a*, and relative refractive index, *m*. The solution of the 'direct problem', can be expressed by Mie'scattering coefficients:

$$a_n = \frac{m\psi_n(mx)\psi'_n(x) - \psi_n(x)\psi'_n(mx)}{m\psi_n(mx)\xi'_n(x) - \xi'_n(x)\psi'_n(mx)} \quad (1.a)$$

$$b_n = \frac{\psi_n(mx)\psi'_n(x) - m\psi_n(x)\psi'_n(mx)}{\psi_n(mx)\xi'_n(x) - m\xi_n(x)\psi'_n(mx)} \quad (1.b)$$

where ψ_n and ξ_n are the Riccatti-Bessel functions. The size parameter $x = 2\pi\, n_s a/\lambda$ is a function of the particle ratio, *a*, and of the incident beam wavelength in vacuum, λ. The optical constants of the material, i. e., the scattered index and the absorption index, become respectively the real and the imaginary part of the particle refractive index, n_p. Denoting as n_s the solvent refractive index then, the relative refractive index is $m = n_p / n_s$.

If $m \to 1$, a_n and b_n vanish. The particle has disappeared, and the light is not scattered.

For the range of wavelengths of the incident beam commonly used in the experimental equipments, the absorbed light by the polymer particles can be neglected for the materials that we are considering. For this reason in this work *m* is approximated by its real value.

The scattered field is established in all directions. It can always be specified as a combination of the two independent scattering amplitudes (Bohren and Huffman (1883) [12]) S_1 and S_2,

$$S_1 = \sum_n \frac{2n+1}{n(n+1)}\left(a_n\pi_n + b_n\tau_n\right),$$

$$S_2 = \sum_n \frac{2n+1}{n(n+1)}\left(a_n\tau_n + b_n\pi_n\right). \quad (2)$$

For example, for an unpolarized incident beam, the differential scattering cross section for a spherical particle of diameter $D=2a$ is given by:

$$S_{11}(\theta, D, m) = \frac{1}{2}\left(|S_1|^2 + |S_2|^2\right). \quad (3)$$

In eqns. (2), τ_n and π_n depend on the scattering angle θ, and are given by:

$$\pi_n = \frac{P_n^1}{sen\theta}; \quad \tau_n = \frac{dP_n^1}{d\theta}; \quad P_n^1 = -\frac{dP_n}{d\theta}; \qquad (4)$$

$P_n^{\ j}$ stands for the associated Legendre functions of the first kind. The n-order Legendre polynomial satisfies the equation:

$$\frac{d}{d\theta}\left(\sin\theta \frac{dP_n}{d\theta}\right) = -n(n+1)P_n \sin\theta \quad .$$

The intensity of the scattered field in a point at a large distance from the particle, *r*, is related to the scattering amplitudes through S_{11}, and to the intensity and wavelength in the medium of the incident beam (I_0 and λ) by

$$I_s = \frac{S_{11}}{k^2 r^2} I_0 . \qquad (5)$$

The intensities ratio is in fact the intensity measured at the detector, which is clearly proportional to the differential scattering cross section:

$$I = I_s / I_0 = c_o S_{11} . \qquad (6)$$

When the material is composed by a collection of spheres of different sizes, the intensity of the scattered light in a point is the superposition of the intensities scattered by each particle. In the limit of the continuous distribution *f(D)* represents the PSD, with *f(D) dD* particles with diameter between *D* and *D+dD* per unit volume. Then, the total scattering intensity is calculated as

$$I(\theta, m) = \int_{D_1}^{D_2} S_{11}(\theta, D, m) f(D) dD . \qquad (7)$$

The scattering measurements in this work are denoted by $g(\theta)$, which is represented mathematically by the scattered intensity obtained for the actual particle refractive index, m_0, plus the measurement noise, $\varepsilon(\theta)$, i. e.

$$g(\theta) = I(\theta, m_0) + \varepsilon(\theta) \qquad (8)$$

INVERSION OF LIGHT SCATTERING DATA

In practice, it often occurs that the particles responsible for the scattering cannot be analyzed directly. From a study of the scattered field, we have to determine the characteristics of the particles, which are responsible for the scattering. This is the 'hard' problem, which is unfortunately the most frequently encountered one.

Inversion of light scattering from a sphere can be defined, as any procedure by which the original data set is reduced to a refractive-index value or profile, according to the homogeneity of the particle. Inversion of light scattering from a suspension of homogeneous spheres is a procedure by which $g(\theta)$ is used to retrieve the particles refractive index and the suspension PSD.

The Problem stated as a minimization problem with a least square functional

The problem is to find the particle size distribution (PSD) denoted as *f* and the refractive index *m* corresponding to the observed data $g(\theta)$ of the total scattering intensity.

To present the mathematical formulation of the problem first we introduce the following notation; let *T* be the operator from the space $L^2[(D_m, D_M)]$ into $L^2[(\theta_m, \theta_M)]$, defined by

$$T_m[f](\theta) = \int_{D_m}^{D_M} S_{11}(\theta, D, m) f(D) dD = I(\theta, m) . (9)$$

With the above notation the stated problem consists on finding a function *f* and a scalar *m* such that $m \varepsilon [M_0, M_1] \subset \Re$ and

$$T_m[f](\theta) = g(\theta). \qquad (10)$$

For each *m*, this problem (eq. (10)) has a solution only if the function *f* is on the image of the operator T_m or in the boundary of this set. We use the familiar notation $\overline{R(T)}$ for this set and its boundary.

Due to the fact that $g(\theta)$ is a measured quantity (it has noise), in general it will not be in $\overline{R(T)}$. Therefore we look for a generalized solution: a solution of eq. (10) in the least square sense. Thus, we state the problem as the minimization of the following objective functional:

$$J(m, f) = \int_{\theta_m}^{\theta_M} \left(T_m[f](\theta) - g(\theta)\right)^2 d\theta \qquad (11)$$

If J is a convex functional of *(m,f)* then its minimum exists and is unique. This cannot be assured for the general case of functional (11). On the other hand, when m is known and the only unknown is the function f, the least square functional will be convex since the inverse problem is linear; this fact is clear from eq. (9). Thus, the existence of the solution of the minimization problem stated in eqn. (11) will be determined by the dependence of kernel $S_{11}(\theta, D, m)$ on m.

In what follows we will discuss the existence of a solution of our inverse problem, which is the existence of a minimizer (m_0,f_0) of $J(m,f)$.

To begin, we deal with each unknown separately. First, we assume that the distribution f is known. Then, the scattering measurements must be inverted to obtain the refractive index. In this case J is a function of m. We will study the direct problem only for the parameter m to determine the existence of a minimum of (11). In this case, the functional has the simpler form:

$$J(m) = \int_{\theta_m}^{\theta_M} \left(I(\theta, m) - g(\theta)\right)^2 d\theta \ . \qquad (12)$$

The possible local minima must satisfy the equation $\frac{\partial}{\partial m} J(m_0) = 0$. It is possible to write the derivative with respect to m as

$$J'(m) = 2\int_{\theta_m}^{\theta_M} (I(\theta, m) - g(\theta))\frac{\partial I(\theta, m)}{\partial m} d\theta \quad . (13)$$

Let's first assume that we have noise-free measurements. Then, $\varepsilon(\theta) = 0$ in eq. (8) and $I(\theta, m_0) = g(\theta)$, so

$$J'(m) = 2\int_{\theta_m}^{\theta_M} \left(I(\theta, m) - I(\theta, m_0)\right)\frac{\partial I(\theta, m)}{\partial m} d\theta \quad (14)$$

We observe that for most of the polymeric emulsions with which we are concerned, there is a unique global minimum at $m=m_0$. The reason is that $I(\theta, m)$ is an injective function on m. We can prove this as follows.

Let's assume that $I(\theta, m)$ is increasing on $m \; \forall \; m \varepsilon \left[M_0, M_1\right]$. Then, for $m>m_0$,

$$\left(I(\theta, m) - I(\theta, m_0)\right) > 0 \; and \; \frac{\partial I(\theta, m)}{\partial m} > 0 \ ,$$

making the integrand positive $\forall \; \theta \; \Rightarrow J'(m) > 0$.

Now, for $m<m_0$,

$$\left(I(\theta, m) - I(\theta, m_0)\right) < 0 \; and \; \frac{\partial I(\theta, m)}{\partial m} > 0 \Rightarrow J'(m) < 0.$$

In consequence, since $J'(m) > 0$ for $m>m_0$ and $J'(m) < 0$ for $m<m_0$, the unique minimum in $\left[M_0, M_1\right]$ is $m=m_0$ .Similarly, if $I(\theta, m)$ is decreasing on $m \; \forall m \varepsilon \left[M_0, M_1\right]$, then $\left(\frac{\partial I(\theta, m)}{\partial m} < 0\right)$.

So, for $m<m_0$, $J'(m) < 0$, since $\left(I(\theta, m) - I(\theta, m_0)\right) > 0$, and for $m>m_0$, $J'(m) > 0$, since $\left(I(\theta, m) - I(\theta, m_0)\right) < 0$.

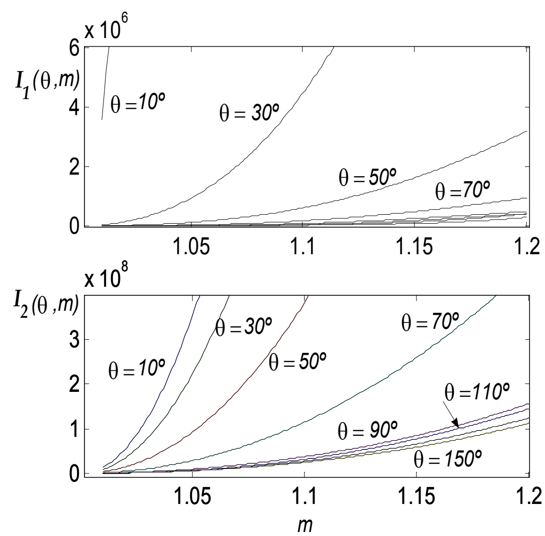

Figure 1. Relation between ELS Intensity and refractive index, for each scattering angle. Simulations corresponding to emulsions with different PSDs.

The condition of being $I(\theta, m)$ increasing on m, $\forall \; m \varepsilon \left[M_0, M_1\right]$, is fulfilled for most of the monodispersed and polydispersed polymeric emulsions we deal with. This was verified numerically for different cases. We show first in Figure 1 $I_1(\theta, m)$ and $I_2(\theta, m)$ obtained for two polydispersed polymeric emulsions. The different PSDs are shown in Figure 2; they are denoted by $f_1(D)$ and $f_2(D)$ respectively.

Notice that the range in which we explore the relative refractive index is $1.0<m<1.2$ as reported for emulsions of polymers and copolymers in the literature [13-14] for wavelengths in the range of 400 to 840 nm. Notice also that *m* is considered real since absorption can be neglected for these emulsions at those wavelengths.

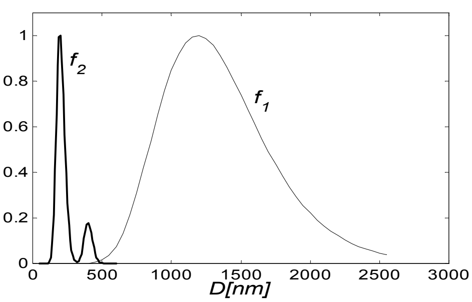

Figure 2. PSD of two polystyrene emulsions

Figure 3 shows $I(\theta, m)$ vs. m for many mono-dispersed emulsions. For suspended particles with D<2500nm, the injective condition holds for any θ. For larger particles this condition is not verified for small angles. For example, for particles with D=2500nm, $I(\theta, m)$ is injective on m only for θ>30º.

We developed computer programs to simulate the experiments. The program used to calculate scattered intensities is based on a program taken from Bohren and Huffman's book [12]. A similar program is also reported in [15].

To deal with the other unknown, i. e. the function *f*, we assume that *m* is known to be m_o. Then, the scattering measurements must be inverted to obtain the PSD, *f*. In this case *J* is a function of *f*,

$$J(f) = \int_{\theta_m}^{\theta_M} \left(T\,[f](\theta) - g(\theta)\right)^2 d\theta \ . \qquad (15)$$

This functional is convex, since *T[f]* is linear on f (eq. (1)). The equation

$$J'(f) = 0\,, \qquad (16)$$

has a unique solution given explicitly by the well-known generalized solution [16]

$$f_o = \left(T^*T\right)^{-1} T^* I(\theta, m_o) \qquad (17)$$

for noise-free measurements, as long as $\left(T^*T\right)$ has positive eigenvalues.

We return now to the originally formulated inverse problem, in which we must consider the two unknowns m and f simultaneously. We want to determine the existence of a minimizer (m_0,f_0) of $J(m,f)$, which must satisfy the conditions:

$$\frac{\partial}{\partial m} J(m,f) = 0 \qquad (18.a)$$

$$\frac{\partial}{\partial f} J(m,f) = 0 \ , \qquad (18.b)$$

where $J(m,f)$ is given by eq. (11). The derivative (18.b) must be considered in a functional sense, for example as a Fréchet or Gateaux derivative.

The conditions obtained when we analyzed the

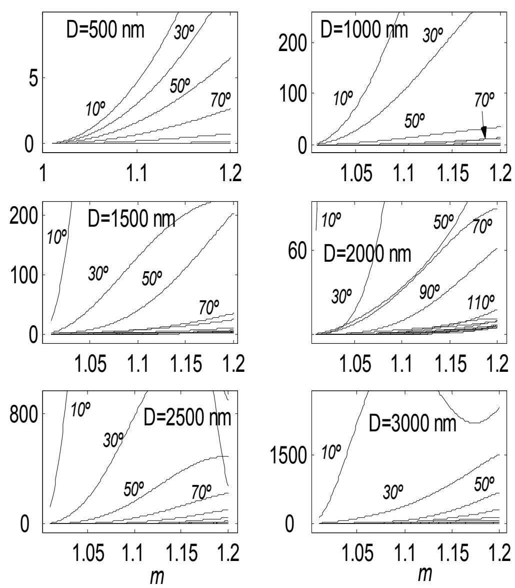

Figure 3. Relation between the Light Scattered Intensity and the refractive index, for each scattering angle. Simulations corresponding to monodispersed emulsions.

existence of a minimizer for each unknown separately are not enough to assure uniqueness of the solution of the equation (18). Instead, additional requirements must be established.

We consider first eq. (18.b). For any fixed *m* the situation is similar to that discussed for eq.(16), so we can assure that its solution exists, it is unique and it is given by

$$f(m) = \left(T(m)^* T(m)\right)^{-1} T(m)^* I(\theta, m_o) \qquad (19)$$

Now, instead of considering eq.(18.a) we proceed to substitute eq.(19) into functional (11).

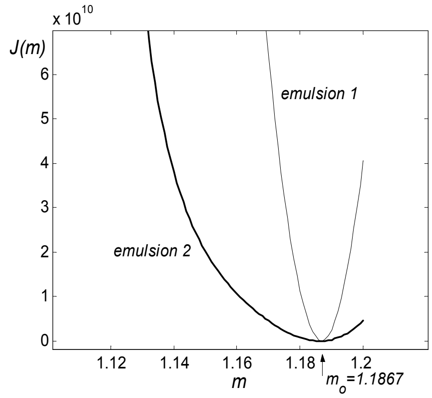

Figure 4. Cost functional as in eq.(20) for two polystyrene emulsions with PSDs shown in Fig2

Then, we obtain for noise free measurements

$$J(m) = J(m, f(m)) = \\ = \int_{\theta_m}^{\theta_M} \left(T_m\left[f(m)\right](\theta) - I(\theta, m_0)\right)^2 d\theta \qquad (20)$$

The condition for the minimum is written now as

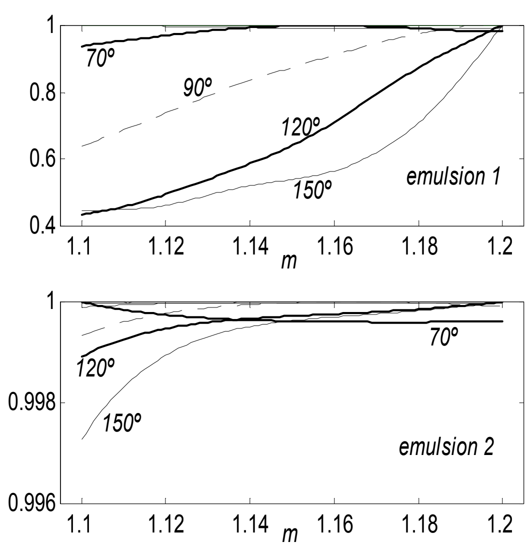

Figure 5. $T_m\left[f(m)\right](\theta)$ vs. m for various values of θ calculated numerically for two emulsions (shown in Fig.2)

$$\frac{dJ(m)}{dm} = 0$$

$$\int_{\theta_m}^{\theta_M} \left(T_m\left[f(m)\right] - T_{m0}\left[f(m_0)\right]\right) \frac{dT_m\left[f(m)\right]}{dm} d\theta = 0. \quad (21)$$

Following the same reasoning that we developed for the simplest case treated in eq. (13), we can affirm that the minimum would be unique at $m=m_o$ if $T_m\left[f(m)\right]$ is injective on m, for any θ. This sufficient condition is in fact very strong.

For the polymeric emulsions we deal with, we showed by numerical simulations that this sufficient condition may not be verified for all θ. However, the cost functional *J(m)* (eq.(20)) still has a unique minimum at m_0, as in Figure 4. To obtain this figure we generated synthetic ideal measurements for the same two polystyrene emulsions (m_0 =1.1867) with PSDs as shown in Figure 2. Figure 5 shows the plots obtained for the relations $T_m\left[f(m)\right](\theta)$ vs. m for different values of θ. For the numerical simulations we must use the discrete version of the inverse problem, in which case, the operation $T_m\left[f(m)\right]$ is aproximated, for each value of m, by a product of a matrix **A** by a vector **f** . Vector **f** is the discrete version of eq.(19), given by $\mathbf{f} = \left(\mathbf{A}^T \mathbf{A}\right)^{-1} \mathbf{A}^T * \mathbf{I}(\theta, m_o)$. See Ref. [21] for more details.

Noisy measurements

We consider now the more realistic case in which the observations are perturbed by an additive noise.

We denote by $g_\varepsilon(\theta)$ the model for the noisy measurements

$$g_\varepsilon(\theta) = I(\theta, m_0) + \varepsilon(\theta)$$

and substitute it in eq.(13) for the case where f is assumed known and we want to determine m. It yields

$$J'(m) = 2\int_{\theta_m}^{\theta_M} \left(I(\theta, m) - I(\theta, m_0) - \varepsilon(\theta)\right) \frac{\partial I(m,\theta)}{\partial m} d\theta \quad =$$

$$= 2\int_{\theta_m}^{\theta_M} \left(I(\theta,m) - I(\theta,m_0)\right)\frac{\partial I(m,\theta)}{\partial m}\,d\theta - \underbrace{-2\int_{\theta_m}^{\theta_M} \varepsilon(\theta)\frac{\partial I(m,\theta)}{\partial m}\,d\theta}_{G(m)} \quad (22)$$

The facts analyzed before for $\varepsilon(\theta) = 0$ hold for the first term in eq.(22). The second term shows that the minimum will change in the presence of noise. However, since $I(m,\theta)$ is a smooth function and $\varepsilon(\theta)$ can be considered a zero-mean uncorrelated random process, then $G(m) \approx 0$. The shift produced by the presence of noise will barely affect the determination of parameter m from the equilibrium equation, $J'(m)=0$. This is shown

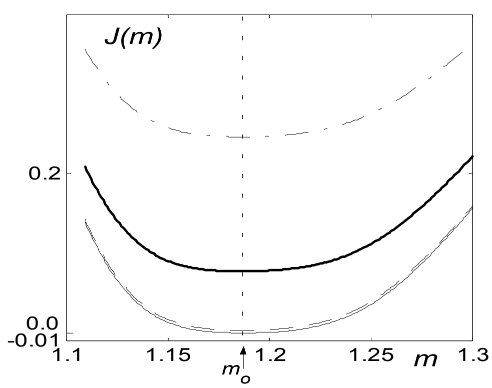

Figure 6. Computation of eq.(22) for simulated ELS measurements for emulsion 1with different noise levels: —— $\varepsilon(\theta)$=0; ---- σ_ε=1%; —— σ_ε=5%; -·-·- σ_ε=10%;

numerically in Figure 6 for the case of emulsion 1 for different noise level. The noise was introduced as a normally distributed white noise with standard deviation σ_ε, which value is given as a percentage of the maximum measurement. This result shows that the solution is stable and so the inversion of ELS measurements to determine refractive index is a well-conditioned problem.

When we assume that m is known to be m_o and f must be determined, the minimum for functional (15) is obtained introducing the noise in eq. (17), given:

$$f_\varepsilon = (T^*T)^{-1}T^*\left(I(\theta,m_o) + \varepsilon(\theta)\right)$$
$$= (T^*T)^{-1}T^*I(\theta,m_o) + (T^*T)^{-1}T^*\varepsilon(\theta)\ .$$

Then,

$$f_\varepsilon = f_0 + (T^*T)^{-1}T^*\varepsilon(\theta) \quad (24)$$

For ill-conditioned operators, which is usually the case for ELS measurements, the solution f_ε may differ greatly from f_0 even for a small perturbation $\varepsilon(\theta)$, since $(T^*T)^{-1}$ amplifies the noise effect. The methodology usually used to obtain a stable solution such that $\|f_\varepsilon - f_0\| < \|\varepsilon\|$, consists in modifying the operator (T^*T) by a well-conditioned one,

$$f_{\varepsilon,\gamma} = (T^*T + \gamma L)^{-1}T^*\left(I(\theta,m_o) + \varepsilon(\theta)\right) \quad (25)$$

known as Phillips-Tikhonov regularization [17,18], since $(T^*T + \gamma L)$ for $\gamma>0$ have positive eigenvalues. L is usually the identity operator, but can also represent the first or second derivative; this is done in order to impose on f the condition of being smooth. γ is the regularization parameter that can be selected, for linear problems, by different methods [19, 20,21].
For the complete problem where m and f must be determined simultaneously, eq. (25) may be written for each m as:

$$f_{\varepsilon,\gamma}(m) = (T(m)^*T(m) + \gamma L)^{-1}T(m)^*g(\theta) \quad (25)$$

and replaced into the functional

$$J(m,f) = \int_{\theta_m}^{\theta_M} \left(T_m[f](\theta) - g(\theta)\right)^2 d\theta\ . \quad (26)$$

in which case we obtain

$$J(m,f) = J(m, f_{\varepsilon,\gamma}(m)) = J(m)$$

as we did before for the noise-free case. The sufficient condition for the minimum of $J(m)$ to be unique can be expressed as a condition on $T_m[f_{\varepsilon,\gamma}(m)](\theta)$, i.e. to be injective on m, for any θ, similarly to that obtained before for the noise-free case. The regularization level applied would be important for the estimation of the PSD. We have proposed for this particular problem [11] an iterative procedure to select the value of the regularization parameter, based on the generalized cross validation technique [19].

CONCLUSIONS

We have analyzed the inversion of ELS measurements to determine PSD and refractive index. We considered polymeric emulsions, composed by homogeneous, spherical and non-absorbing particles.

The mathematical formulation results in a nonlinear inverse problem. A Fredholm integral equation of the first kind appears with an unknown parameter in its kernel.

We stated a minimization problem with a least square functional and analyze the generalized solutions obtained. We considered first each unknown independently, resulting in two separate problems; the inversion of ELS for a given PSD to obtain the refractive index, and the inversion of ELS for a given refractive index to obtain the PSD. This second problem is linear so it is completed covered by the well-developed theory of inverse problem.

The first problem is nonlinear. We obtain a sufficient condition to assure uniqueness of the solution. The relation between the measurements and the parameter must be injective. We have found that this is the case in most of polymeric emulsions.

For noisy measurements, and because of the ill-posed nature of the inverse light scattering problem, Phillips-Tikhonov regularization form is proposed to stabilize the problem. We proved that no regularization is necessary for the refractive index and that a high accuracy calculated value will be obtained for this parameter from scattering data with typical error level.

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