

EXPERIMENTAL CHARACTERIZATION OF THERMAL RADIATION PROPERTIES OF DISPERSED MEDIA

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ABSTRACT

As dispersed materials generally are semi-transparent media which absorb, emit and scatter thermal radiation, the predictive modeling of thermal processes involving such kind of materials requires the knowledge of a number of radiative properties to feed the models. These properties cannot be directly measured but are identified from a set of experimental data of radiative flux collected from a sample submitted to appropriate experimental conditions. This paper focuses on identification methodology for thermal radiation properties of dispersed media such as fibers, foams, pigmented coatings, ceramics.. After a brief introductory overview of the subject, the parameter identification methodology and two experimental facilities used for radiative properties determination are firstly described. As the identification process involves a solution model for the Radiative Transfer Equation inside the sample, some attention is then paid to the development of RTE solution models well matched to this specific purpose. Two examples of application are described before concluding on the advantages, limitations and remaining difficulties connected to this new and promising metrology of thermal radiative properties of dispersed media.

NOMENCLATURE

CN	matrix condition number
i_λ	spectral intensity of radiation
$f_{1,2}$	weights in a composed phase function
g_λ	parameter of Henyey-Greenstein phase function
i_λ	spectral intensity of radiation
$i_{b\lambda}$	spectral intensity of blackbody radiation
N	number of measurement data
N_{bd}	number of bi-directional measurement data
nd	number of discretization points of the quadrature
p_j	jth parameter to be identified
P_λ	scattering spectral phase function
R_h	hemispherical reflectance
T	transmittance or reflectance
T_{bd}	bi-directional transmittance or reflectance
T_h	hemispherical transmittance
$X(i,j)$	normalized sensitivity coefficient

Greek symbols

β_λ	spectral volumetric extinction coefficient
κ_λ	spectral volumetric absorption coefficient
μ	$\cos(\theta)$
θ	polar angle of direction
θ_0	incident and scattering directions angle
σ_λ	spectral volumetric scattering coefficient
τ_0	optical thickness
ω_λ	spectral albedo

Subscripts

b	blackbody
e	experimental data (measured)
t	theoretical data (computed)
λ	wavelength

INTRODUCTION.

A number of materials widely used in many industrial areas such as aerospace, textile, building, food, energy and environment.., are dispersed and semitransparent media which emit, absorb, and scatter thermal radiation. Heat transfer by combined radiation with conduction or convection in such media is a problem of great practical importance, mainly in situations where radiation is the dominant mode. Most of these materials are composed of dispersed phases of solid/solid type (ceramics, surface pigmented coatings..), or solid/gas type (thermal insulation materials : fibers, foams, fluidized and packed beds, catalytic combustors, soot..), or liquid/gas type (sprays..).

The prediction of radiative heat transfer in semitransparent materials requires not only the capability to solve the Radiative Transfer Equation (RTE) but also the knowledge of the radiative properties of the concerned media.

Although numerical and analytical methods are available to solve the RTE for dispersed media, some difficulty remains to determine the radiative properties of such media and there is a need of appropriate methodologies. This paper will be focused on the determination of the radiative properties of dispersed media.

The properties of which knowledge is required are the refractive index n_λ , the absorption coefficient κ_λ and the scattering coefficient σ_λ , or alternatively the extinction coefficient $\beta_\lambda = \kappa_\lambda + \sigma_\lambda$ and the albedo $\omega_\lambda = \sigma_\lambda / \beta_\lambda$, and the phase function P_λ . This means that at least four spectral properties should be determined and even more as P_λ is defined from several shape parameters.

The reviews of Viskanta and Mengüç [1], and, more recently, Baillis and Sacadura [2] report a number of works devoted to radiative transfer in dispersed media and radiative properties of such media over the last 20 years.

Basically there are two families of methods allowing the determination of thermal radiation properties: (i) the predictive modeling, and (ii) experimental methods using a parameter identification technique applied to some appropriate set of radiative flux measurement data.

i) As reported in Baillis and Sacadura in [2], a significant number of works have been realized on theoretical prediction of radiative properties of fibrous media or media composed of spherical particles, starting from the properties of the basic components such as the optical indices. The modeling method mostly used is based on the Mie's theory describing the interaction of an electromagnetic wave with an individual particle. But in case of foams or ceramics, few works are found due to the complexity of these media. Measurement data of spectral transmittances and reflectances are frequently used either in order to compare experimental data to theoretical results, or to identify unknown properties of the basic components, for instance the hemispherical reflectance of the solid phase of the foam [3]. The main interest of these models is that they allow to study the influence of porosity and of the radiative properties of the basic components of the media, and they also contribute to improve the understanding of the phenomenological aspects.

ii) An alternative way to determine the radiative properties of a medium consists of identifying them. It allows to study complex materials of which radiative behavior would not be easily modeled from theory. Two types of transmittance and reflectance measurements may be used to provide the data for the identification process: directional-hemispherical or directional-directional (bi-directional) measurements. A number of works have been published dealing with radiative properties identification based on directional-hemispherical experimental data, and assuming an isotropical phase function. Among the more recent ones are the works of Skocypec et al. [4], and Hale and Bohn [5] on reticulated ceramics, and Kuhn et al. [6] on polystyrene and polyurethane foams. Hendricks and Howell [7] also identified the radiative properties from hemispherical transmittance and reflectance measurements, but they used non isotropical phase functions. Four parameters have been so identified by these authors: the absorption and scattering coefficients and two coefficients for the phase function. In order to get enough information to carry out the identification procedure, they used samples of different thicknesses. Nicolau et al [8] used directional-directional measurements to identify the radiative properties of fibrous insulating materials: extinction coefficient, albedo, phase function parameters. A new phase function representation as been proposed by these authors which combines two Henyey and Greenstein phase functions, forward and backward peaked, respectively, coupled with an isotropic phase function. Moura et al. [9] used directional-hemispherical measurements, but considering different angles of incidence onto the sample in order to increase the amount of information. They identified the same parameters for the same materials as in [8], (the phase function representation was also the same). These authors have shown through numerical simulations that bi-directional measurements with a collimated beam normally incident onto the sample are more appropriate than directional-hemispherical transmittance and reflectance measurements under different angles of incidence to identify the radiative properties of this kind of media. Recently Milandry [10] identified the radiative properties of silica wool by using a bi-directional experimental set-up. In order to obtain a phase function closer to the one given by Mie's theory he added a Lorentz function to the Nicolau's phase function, which means one more parameter to identify.

However, like in case of Nicolau's phase function, it appeared that there are too much parameters to identify, mainly when some of them are correlated.

Both measurement methods, directional-hemispherical or directional-directional with normal incidence show advantages and drawbacks. Directional-hemispherical measurements are easily and rapidly acquired, but they only allow the identification of the extinction coefficient and albedo for a given sample thickness, the phase function being assumed known. On the other hand directional-directional measurements contain much more information allowing the identification of parameters of the phase function, but for the directions of incidence other than those situated close to the normal the measured data are weak and noised. Furthermore the phase function remains difficult to identify [8]. For that reason Baillis et al. [11] used a combination of directional-directional and directional-hemispherical transmittance data acquired under normal incidence to identify the radiative properties of polyurethane foam.

Other methods are available such as the *flash method* recently extended [12, 13] to simultaneously determine conductive and radiative properties. The main advantage is that they provide the properties as a function of temperature, but they do not allow to obtain spectral properties (only gray ones) and not the phase function. So this lecture will be focused on identification of spectral radiative properties of condensed semi-transparent media from spectral radiative measurements.

In what follows the identification methodology based on the comparison of theoretical and experimental data will firstly be recalled. Different experimental configurations (hemispherical or directional measurements or combination of both types) will be presented. The key role of the sensitivity coefficients and of the condition number of the matrix of sensitivity coefficients will be pointed. Then examples of experimental set-ups will be described, and models allowing to calculate transmittances and reflectances as well as different models of representation of the phase function will be presented. Finally some examples of application of radiative properties determination of fiber and foam materials will be given before concluding.

PARAMETER IDENTIFICATION

Principle

The parameter identification method is based upon the use of:

- a set of experimental data of spectral radiative fluxes (T_{ei}) coming in different directions (i) from one or both sides of a sample irradiated by a source or emitting radiation,
- the same number of theoretical fluxes (T_{ti}) calculated for the same conditions as the experimental data from an initial guessed set of values of the parameters p_1, \dots, p_n to be identified (Fig.1).

For each wavelength the goal is to determine the set of parameter values p_1, \dots, p_n which minimizes the functional F , which represents the sum of quadratic differences between the theoretical and the experimental data:

$$F(p_1, \dots, p_n) = \sum_{i=1}^N [T_{ti}(p_1, \dots, p_n) - T_{ei}]^2 \quad (1)$$

N is the number of experimental or theoretical data. For: *hemispherical measurements*: $N=2$ (only one transmittance and one reflectance measurements),

directional measurements : $N=N_{bd}$, N_{bd} being the total number of directions of bi-directional measurement,
any combination of directional and hemispherical measurements : $N = 2+N_{bd}$.

Several methods are available to achieve the minimization. Uny [14] used the method of Hooke and Jeeves. Nicolau et al. [8], and Baillis et al. [3] adopted the method of linearization of Gauss (see for instance Beck and Arnold [15], which allows to better follow the convergence of the process by calculating at each step a set of sensitivity coefficients. The principle of this method is briefly recalled.

Gauss linearization method

It involves the solution of the following system of non linear equations :

$$\begin{bmatrix} \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_1} \right)^2 & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_1} \right) \left(\frac{\partial T_{ii}}{\partial p_2} \right) & \dots & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_1} \right) \left(\frac{\partial T_{ii}}{\partial p_n} \right) \\ \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_2} \right) \left(\frac{\partial T_{ii}}{\partial p_1} \right) & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_2} \right)^2 & \dots & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_2} \right) \left(\frac{\partial T_{ii}}{\partial p_n} \right) \\ \dots & \dots & \dots & \dots \\ \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_n} \right) \left(\frac{\partial T_{ii}}{\partial p_1} \right) & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_n} \right) \left(\frac{\partial T_{ii}}{\partial p_2} \right) & \dots & \sum_{i=1}^N \left(\frac{\partial T_{ii}}{\partial p_n} \right)^2 \end{bmatrix}^k \begin{bmatrix} \Delta p_1 \\ \Delta p_2 \\ \dots \\ \Delta p_n \end{bmatrix}^{k+1} = \begin{bmatrix} \sum_{i=1}^N (T_{ii} - T_{ei}) \left(\frac{\partial T_{ii}}{\partial p_1} \right) \\ \sum_{i=1}^N (T_{ii} - T_{ei}) \left(\frac{\partial T_{ii}}{\partial p_2} \right) \\ \dots \\ \sum_{i=1}^N (T_{ii} - T_{ei}) \left(\frac{\partial T_{ii}}{\partial p_n} \right) \end{bmatrix}^k \quad (2)$$

An iterative method of solution is used (Fig. 1):

$$p_j^{k+1} = p_j^k + \Delta p_j^k \quad (3)$$

where Δp_j^k is the increment to add to each unknown parameter p_j at iteration k. The LHS matrix S of Eq.(2) is composed of sensitivity coefficients calculated from the theoretical model. They do not depend on the experimental values
A condition number CN of matrix S is calculated at each step :

$$CN(S) = \|S^{-1}\| \cdot \|S\| \quad (4)$$

$\|S\|$ is the norm of the matrix, defined from the elements S_{ij} as:

$$\|S\| = \max_{i=1,n} \sum_{j=1}^n S_{ij} \quad (5)$$

The condition number provides a detection of possible linear dependences between sensitivity coefficients $\partial T_{ii} / \partial p_j$. The condition number is greater than one. The larger the condition number is, the worse ill-conditioned the system is : small changes in the RHS of equation (3), i.e. in the experimental data, will result in very large change in the solution vector, i.e. the increments Δp_j^k . This means that it will be impossible to simultaneously determine all the unknown parameters.

Poor conditioning occurs when at least two sensitivity coefficients are linearly dependent, or when one is very small or very large compared to the others. Thus the condition number is a very powerful tool to previously investigate the probability of success of the parameter identification procedure and, accordingly, to adapt the methodology.

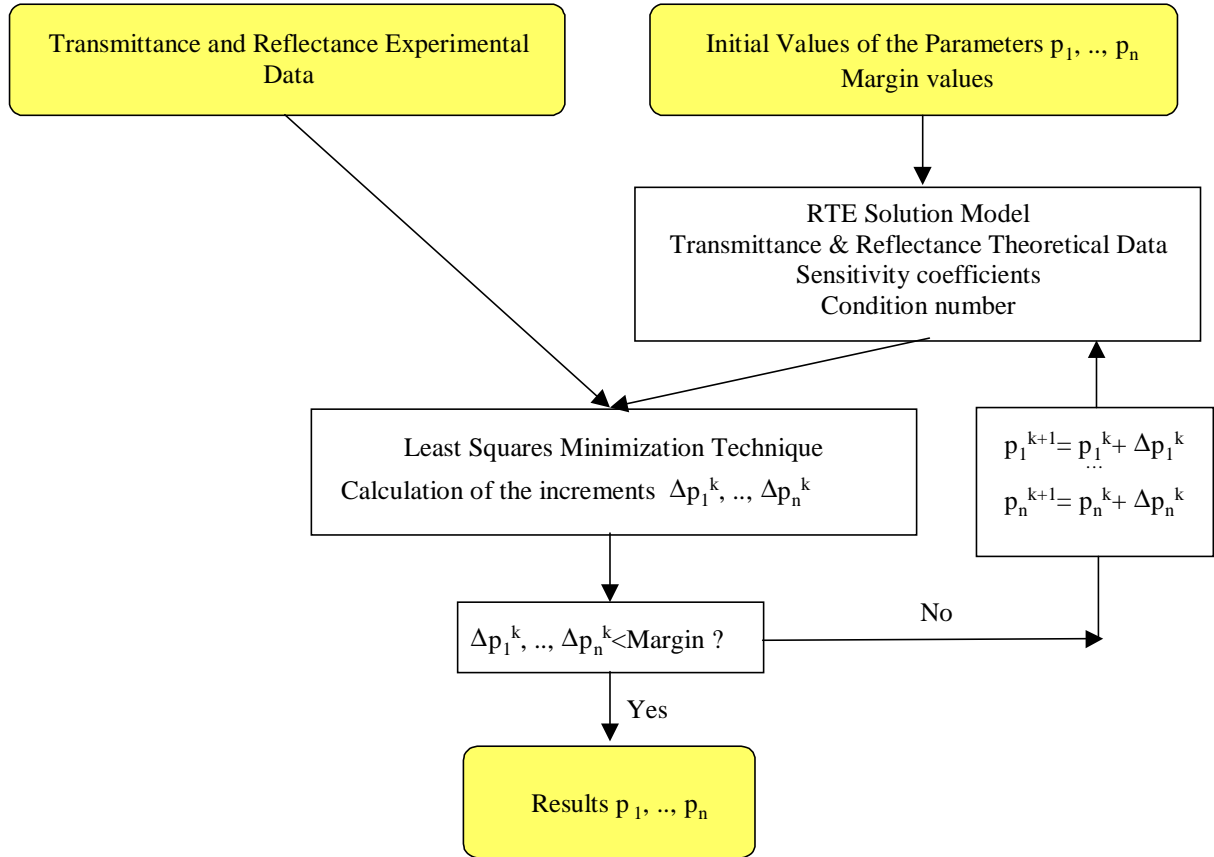


Fig. 1: Flow chart of the parameter identification procedure

unit, with a liquid nitrogen cooled MCT detector. The radiation source of the FTIR is a SiC globalbar. The wavelength range allowed for measurements is [1.4 μm – 18 μm]. The diameter of the sample area submitted to a normally incident beam is of 13 mm.

RTE SOLUTION MODEL

In order to calculate the theoretical data used in the radiative parameter identification process, a very efficient solution method in terms of accuracy/computing time ratio is required for the Radiative Transfer Equation. The RTE, which describes the variation of the spectral radiation intensity i_λ (in a given direction Ω , as a function of optical depth τ) in an absorbing-emitting-scattering medium, can be written as:

$$\frac{1}{\beta_\lambda} \Omega \nabla i_\lambda(\tau, \Omega) + i_\lambda(\tau, \Omega) = (1 - \omega_\lambda) i_{b\lambda}(\tau) + \frac{\omega_\lambda}{4\pi} \int_{\Omega'=4\pi} i_\lambda(\tau, \Omega') p_\lambda(\Omega', \Omega) d\Omega \quad (6)$$

This equation involves the spectral radiative properties of the medium : extinction coefficient β_λ , albedo ω_λ , phase function p_λ . $i_{b\lambda}$ is the Planck's blackbody intensity and $d\Omega$ is an elemental solid angle surrounding the direction Ω . These properties are those of a pseudo-continuous medium equivalent in terms of radiative transport, to the real dispersed material. When transmittance and/or reflectance measurements are used, due to the modulation of the measured radiation by the FTIR system, the emission term in the RTE is not accounted for.

Among different methods available to solve numerically the RTE, the Discrete Ordinates Method (DOM) is commonly considered as the most efficient for radiative parameter identification purposes. In this method the integral term of the RTE is calculated from an angular quadrature over a set directions of discretization. [18]. Thus the integral-differential equation is transformed into a PDE system that may be solved numerically, by finite differences for instance or analytically when possible.

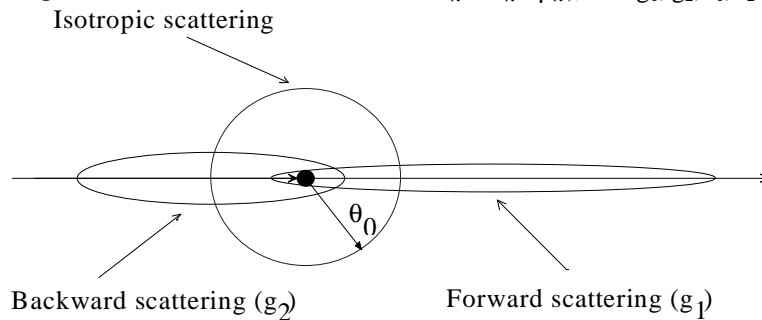


Fig. 4 : Composed HG phase function.

Representation of the phase function

The representation of the phase function, which plays an important role in the RTE solution, should be carefully selected. For media of which particles may be considered as randomly oriented in space, phase function only depends on the scattering angle, that is the angle θ_0 between any couple of incident and scattered radiation directions. A common approach to represent the phase function consists of developing this function in a limited series of Legendre polynomials. Unfortunately the phase functions of a number of particulate media, particularly fibers and foams, exhibit a strong peak in the incident direction (forward scattering) and a fair back-scattering, which requires a very large number of expansion coefficients in a Legendre polynomials expansion. This is of course not suitable for identification. An alternative solution is to use simpler representations of phase function, like Henyey and Greenstein's one (HG), which only needs one shape parameter, g_λ :

$$p_\lambda(\theta_0) = \frac{1 - g_\lambda^2}{(1 + g_\lambda^2 + 2g_\lambda \cos \theta_0)^{3/2}} \quad -1 < g_\lambda < 1 \quad (7)$$

or a combination of different phase functions (Nicolau et al., [8] ; Hendricks and Howell, [7]). As an example, the phase function of Nicolau et al., well suited for fibrous media, may be written as :

$$P(\theta_0) = f_2 [f_1 P_{HG,g_1}(\theta_0) + (1 - f_1) P_{HG,g_2}(\theta_0)] + (1 - f_2) \quad (8)$$

where the parameters g_1 and g_2 govern the shape of the HG functions P_{HG,g_1} and P_{HG,g_2} in forward and backward directions, f_1 is a weigh factor between forward and backward anisotropy of the phase function and f_2 is the weigh factor between anisotropic and isotropic scattering (Fig. 4).

Thus if it is not mandatory to identify the refractive index n_λ of the medium, and by using for instance the Nicolau's phase function, the maximum number of radiative parameters to be identified for a dispersed medium is of 6 . They are: the absorption and scattering coefficients κ_λ and σ_λ (or the extinction coefficient $\beta_\lambda = \kappa_\lambda + \sigma_\lambda$ and the albedo $\omega_\lambda = \sigma_\lambda / \beta_\lambda$), and g_1, g_2, f_1, f_2 .

Non-azimuthal symmetry of the radiative field

Most of available solution methods of the RTE assume azimuthal symmetry of the radiative intensity field. This limits the modeling of the experiments to configurations in which the radiation incident onto the sample is symmetric around the normal to the surface. Moura [17] has recently developed a non-azimuthal symmetric angular quadrature suitable for non-normally incident radiation onto the sample, which increases the number of possible experimental configurations for parameter identification.

EXAMPLES OF APPLICATION

Examples of radiative parameter identification concerning two different dispersed media (fibers and foam) are now presented. They will show the importance of the conditioning number and of the sensitivity coefficients. Both materials were investigated in non emissive situation (cold medium), by using the experimental facilities described above which only work with samples at room temperature.

Bi-directional measurement data

By using the set-up described in Fig. 2, the identification procedure has been applied to a fibrous medium (low density

commercial insulating glass wool). The properties to be determined are the following : $p_{j=1..6} = \omega, g_1, f_1, f_2, g_2, \tau_0$, (in this sequence). The values of the condition number CN show (Fig. 5) that it is difficult to simultaneously identify all 6 parameters. A sample optical thickness of 12 appears as optimal. However as the absorption coefficient is not yet known, it is difficult to select the corresponding geometric thickness for the sample. The CN depends on the number of parameters and on the choice of parameters to identify : in Fig. 5 when only two parameters are identified, these are ω and g_1 ; when three parameters are identified they correspond to the sequence ω, g_1 and f_1 , and so on.. Moreover the analysis of the normalized sensitivity coefficients shows that for $\tau_0 = 2$ the sensitivity of τ_0 remains weak compared to the other parameters. Thus it will be difficult to identify τ_0 for this value of the optical thickness. Conversely, for $\tau_0 = 20$ it is the sensitivity of g_2 which appears to be very low (Fig. 6 a and b). This preliminary investigation of the identification configuration shows that for this material and with this experimental configuration it will be difficult to simultaneously identify 6 parameters. A possible way successfully used by Nicolau is to reduce the number of parameters of the phase function, by assuming for instance $g_2 = -g_1$.

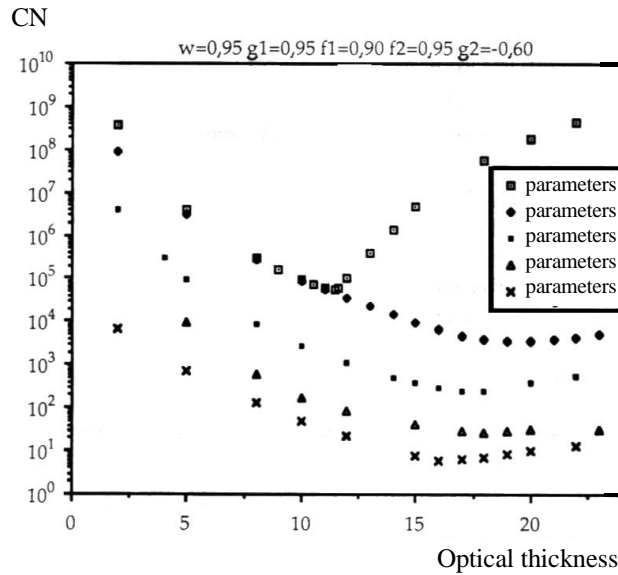


Fig. 5: Condition number as a function of optical thickness.

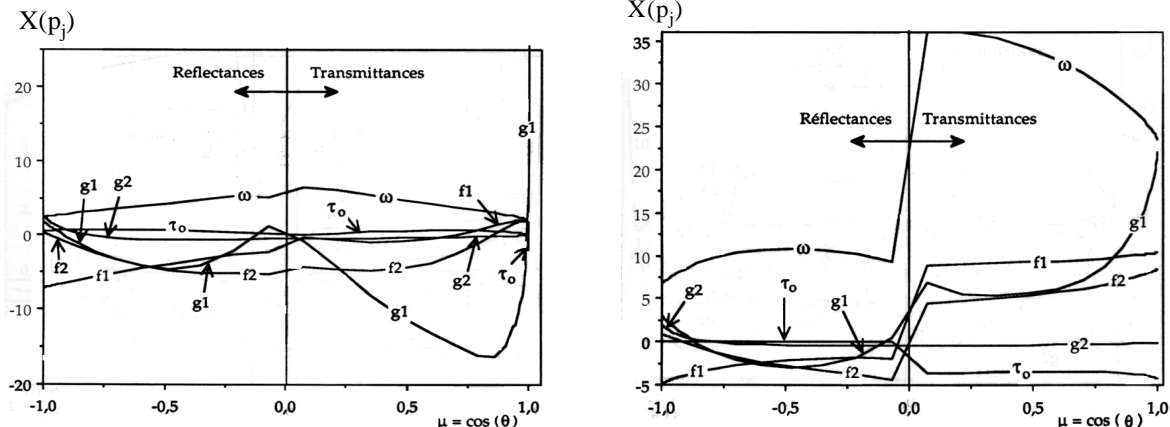


Fig 6: Normalized sensitivity coefficients. $\omega=0.95, g_1=0.95, f_1=0.90, f_2=0.95, g_2=-0.6$. a) $\tau_0=2$, b) $\tau_0=20$.

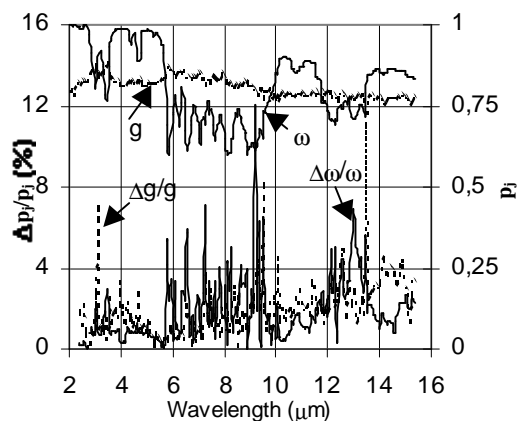
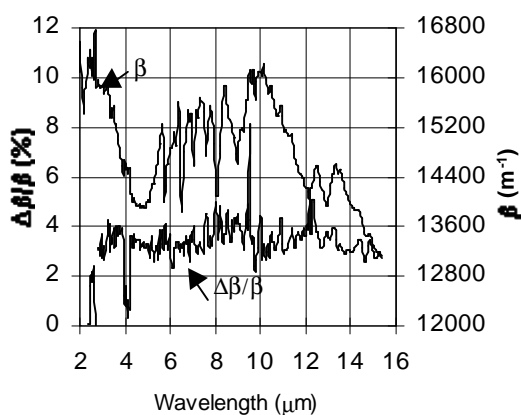
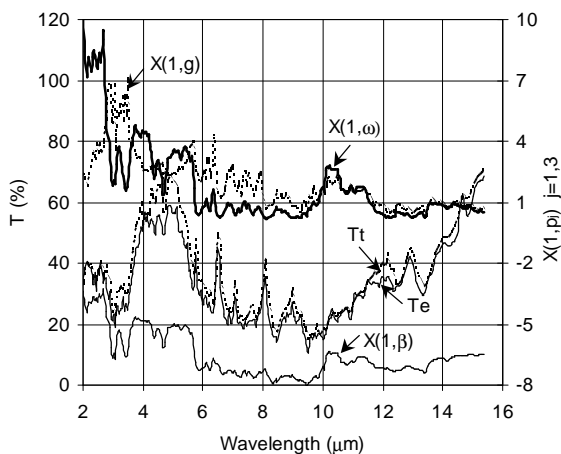


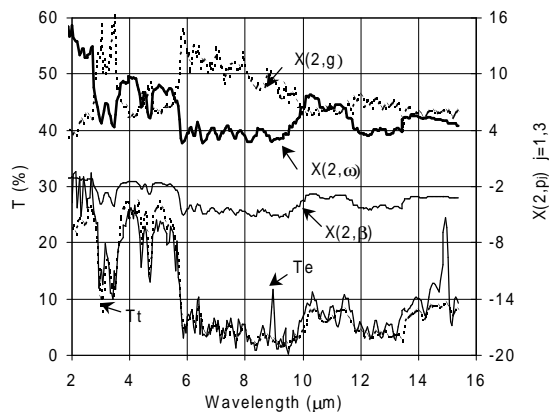
Fig. 7: Average and standard deviation of radiative properties calculated from 3 different thicknesses of sample [11]:

a) Extinction coefficient function.

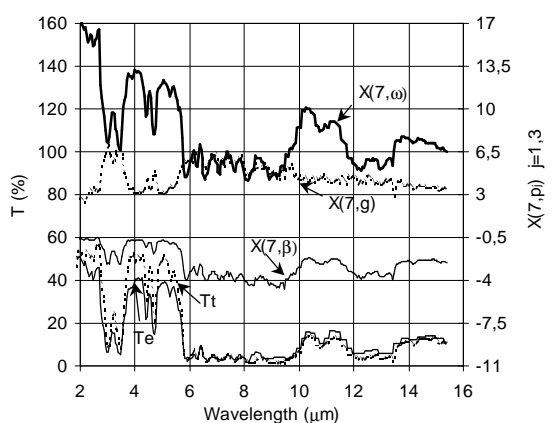
b) Albedo and coefficient of Henyey and Greenstein phase function.



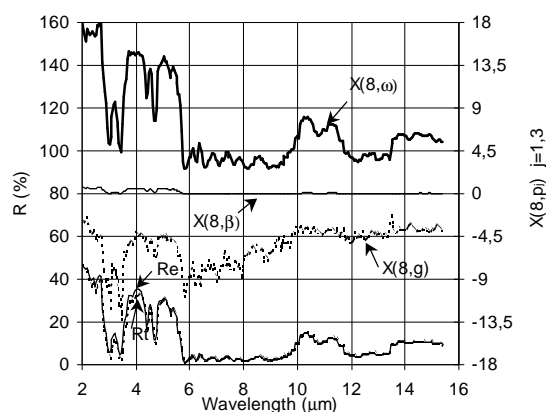
a) Bi-directional transmittance for direction 1 ($\mu=1$).



b) Bi-directional transmittance for direction 2 ($\mu=0.99948$).



c) Hemispherical transmittance.



d) Hemispherical reflectance.

Fig. 8: Normalized sensitivity coefficients of parameters: β_λ , ω_λ , g_λ versus wavelength – Experimental and theoretical results – Sample of thickness 528 μm .

a) Bi-directional transmittance for direction 1 ($\mu=1$), b) Bidirectional transmittance for direction 2 ($\mu=0.99948$), c) Hemispherical transmittance, d) Hemispherical reflectance [11].

Combination of directional-hemispherical and bi-directional measurement data

When bi-directional measurements are carried out on materials like foams which are mostly optically dense, the recorded signals are very low and noised in backward directions as well as in directions far from the incidence one. But on the other hand if these data are not accounted for the identification, there is not enough information to identify the radiative properties, specially the phase function. Thus Baillis and Sacadura [11] identified radiative properties of polyurethane foam by using a combination of directional-hemispherical and bi-directional measurement data. In order to alleviate the number of parameters to identify they adopted a single HG function of phase representation. The facilities used are those described in Fig. 2 and 3. For each wavelength value a set of 8 experimental data was entered in the identification process : 6 bi-directional transmittance measurements acquired in directions near to the incidence one, where the signal is sufficiently strong, plus 1 hemispherical transmittance and 1 hemispherical reflectance. The standard deviations of the radiative properties values identified from three different thicknesses of sample (310 μm , 427 μm , and 528 μm) remain low (Fig. 7 a and b) which shows a good behavior of the model used. The investigation of the sensitivity coefficients (normalised coefficients X) showed (Fig. 8 a to d) that the parameters seem not to be correlated. β is not sensitive for hemispherical reflectance. Conversely, β is the most sensitive parameter for the normal transmittance (direction 1, Fig.8a). In case of hemispherical transmittance and reflectance the sensitivity of ω appears to be the most important one. For bi-directional transmittance in direction 2, situated very close to the incident direction, the most sensitive parameter is g (Fig. 8b). Thus the combination of directional-hemispherical and bi-directional experimental data provides complementary information making possible the identification of the radiative parameters.

DETERMINATION OF THE REFRACTION INDEX

The refractive index is involved in the source term (Planck's function) of the RTE as well as in the radiative boundary conditions. For high porosity media, as a number of fiber or foam materials are, the refractive index of the pseudo-homogeneous medium equivalent, for radiative transport effects, to the real dispersed material, is nearly equal to the index of the fluid filling phase (generally gas, and mostly air, for fibers or foams), which may be known from literature.

The usual methods to determine the refraction index n_λ are based upon the use of reflexion spectra and Kramers-Kronig relations, or the use of ellipsometric techniques. A serious problem occurs in case of dispersed materials, made of particles: it is not possible to perform measurements on individual particles *as they are* in a dispersed medium. In fact the reflexion data are acquired on samples of slab-shape, made of agglomerated particles, obviously not exactly the same material as in the dispersed phase. Once the index of refraction of each dispersed phase and/or the matrix is supposed to be known, an equivalent refractive index of a pseudo-homogeneous medium equivalent for radiative transport to the real dispersed medium, may be calculated from averaging theories like : Maxwell-Garnett (spherical

particles in a dielectric matrix), Maxwell-Garnett modified by Cohen et al. (accounting for non-spherical shape of particles), Bruggeman, Wood & Ashcroft Effective Medium Theory (both shapes are particles, spherical and ellipsoidal) [19;20]. But in fact the accurate determination of the refractive index of dispersed media should be considered today as an unsolved problem, open to research.

CONCLUSION

As shown in this paper experimental parameter identification based on a new and smart metrology matched to each material to be investigated, is a promising way to determine thermophysical radiative properties of complex media. Each medium may be a specific case and requires a preliminary metrological analysis, accordingly to the type of material and the expected accuracy, mainly on the knowledge of the phase function.

The accuracy required on the phase function plays an important role in the selection of the identification strategy : if an isotropic phase function is assumed, the identification is simpler, two hemispherical measurements (transmittance and reflectance), that may be rapidly acquired, are sufficient. Also the selection of the phase function is not independent from the choice of the radiative transfer model using the identified properties : for instance if a highly forward peaked Henyey and Greenstein phase function identified from bi-directional measurements is then used in a rather simplified RTE solution model like the two-flux model (much less accurate than the Discrete Ordinates Method), it is obvious that significant errors may occur. In this case the use of hemispherical measurements and of an isotropic representation for the phase function would be preferable and certainly easier to carry out.

The type of medium to be studied is also important. If it does not allow to measure enough radiative energy in directions far from the normal to the surface of the sample, the acquired information will not be sufficient to identify the radiative properties including the phase function, from bi-directional measurements. In this case a combination of bi-directional and hemispherical measurements would be better suited.

The sensitivity coefficients and the condition number are very helpful tools which allow to previously estimate the probability of success of an experiment designed for parameter identification and to match it accordingly. For each application it is also strongly recommended to use different thicknesses of sample in the identification process in order to check that the final results obtained are independent from the material thickness.

The aim of this paper was to present some tracks open by a new experimental methodology in the difficult area of assessment of thermal radiation properties of dispersed media. Most of current know-how is limited to room temperature property determination. Some properties, as the index of refraction of particulate media, still remain hardly accessible to metrology. These are only two, among other aspects, which are urging challenges for radiative property metrology.

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