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Methodologies for determining thermoplastic films optical parameters at 10.6 μm laser wavelength

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Abstract

A set of simple methodologies is presented so that optical parameters relevant to 10.6 μm CO_2 laser radiation processing of thermoplastics films are obtained. These methodologies are applied to transparent and white polyethylene and transparent polypropylene samples, with thicknesses between 10 and 800 μm. Optical parameters like transmittance, reflectance and absorptance are measured directly with two simple experimental apparatus. With these values, two methodologies are developed in order to obtain the attenuation coefficients. One of the methods relies on curve fitting and shows low uncertainty. The other, based on calculations derived from the Beer–Lambert law, presents dependency on experimental errors for samples with lower thickness, needing further calculation in order to minimize it. The attenuation length and the complex refractive index values are also determined. The resulting data demonstrate the high transparency of thermoplastic samples to 10.6 μm radiation for thicknesses lower than 200 μm, which is significant information for modelling thermoplastics laser processing.

Keywords: Optical parameters of polymers; Thermoplastic films; CO_2 laser radiation; Polypropylene; HD PE; LD PE

1. Introduction

A knowledge of optical parameters is of utmost importance to better understanding of any process that involves laser processing of a material. This is true and applicable to polymer materials in general and to thermoplastics in particular. In fact, as laser technology progressed, its application to polymer processing has also shown noticeable growth [1–5]. In particular, for the thermoplastic industry, the replacement of traditional tools (such as hot knives, ultrasound or hot air) by laser tools has been justified by the possibility of increasing process reproducibility, simplicity of moving parts and increase of productivity [6–9]. Also, being a non-contact and non-contaminant process, its application to the manufacturing of food and medical products is of growing interest [10–13].

In spite of advances in the application of new laser sources, namely diode lasers [14,15], the CO_2 laser is still the most commonly used laser in industry. This laser with wavelength (λ) of 10.6 μm, has long been used in plastics processing, in preference to other lasers with shorter wavelengths, taking in account the fact that the sample's absorptance is less sensitive to the presence of colouration pigments [16]. However, analysis of the optical parameters for thermoplastics has been undertaken mainly for plastics with thicknesses higher than 100 μm. Since many applications use thermoplastic films

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with lower thickness (e.g. plastic bags), correct methodologies must be considered so their parameters can be found with acceptable associated errors.

Among the several optical parameters of a material, absorptance, A, and attenuation length, L, (or the related attenuation coefficient, a_T) are of fundamental importance to the understanding of physical phenomena present during processing. Others like reflectance, R, and refractive index, n, characterize the material in terms of optical behavior in the presence of laser radiation and are also important in the evaluation of the interaction.

Common methods like those based in ellipsometry or polarimetry [17] provide high-precision results, but they are based on cumbersome experimental set-ups. In the next section, a set of simple methodologies, both theoretical and experimental, are described. Their objective is to obtain the above-mentioned optical parameters when 10.6 μm laser radiation is applied to transparent and white thermoplastics.

2. Methodology

The attenuation (or extinction) coefficient, a_T, is defined as the fraction of intensity loss per unit of length for a wave travelling in a transparent medium. Considering a plane wave, the fraction lost when a wave crosses an infinitesimal thickness dh is [18]

$$\frac{dI}{I} = -a_T \cdot dh$$

if this thickness has a finite value then

$$\int_0^h \frac{dI}{I} = -a_T \int_0^h dh$$

or, in the known Beer–Lambert law [18] form

$$I = I_0 \cdot e^{-a_T h} \qquad (1)$$

Then, a_T can be obtained through

$$a_T = \frac{1}{h} \ln \frac{I_0}{I} \qquad (2)$$

where h is the sample thickness, and I_0 and I are, respectively, the incident and transmitted irradiances. So, if the transmittance value,

$$T = \frac{I}{I_0} \approx \frac{P}{P_0} \qquad (3)$$

is known, a_T can be obtained. The method applied to obtain T experimentally was based on the direct measurement of the power transmitted through the sample. Fig. 1 shows the schematic set-up used to acquire this value. The incident laser beam was first divided in two by means of a 50% beam-splitter. This way, the

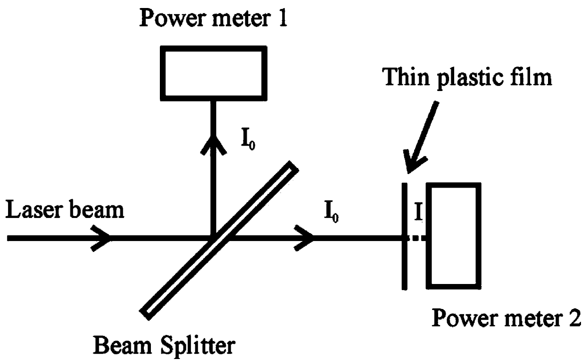

Fig. 1. Schematic set-up for determining transmission values.

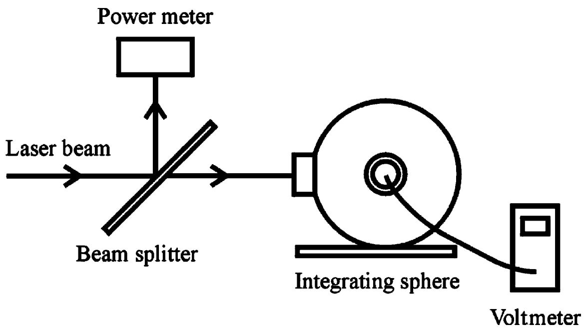

Fig. 2. Schematic set-up for obtaining (R + T) values.

power incident on the sample, P_0, was measured on power meter 1, while power meter 2 measured the transmitted power.

The reflectance of the sample, R, and its absorptance, A, were obtained using an integrating sphere, as shown on Fig. 2. The power meter was used to calibrate the voltage output of the sphere's detector, and also to work as a reference to the incident power on the sample. Using the sphere, the combined value of $R + T$ was obtained. Since the previously acquired data proved that the samples show high transparency (as will be shown below), a frame holder was used for this procedure, allowing both reflectance and transmittance of the thermoplastic to be obtained by the integrating sphere. Fig. 3 shows the frame used to support the thermoplastic

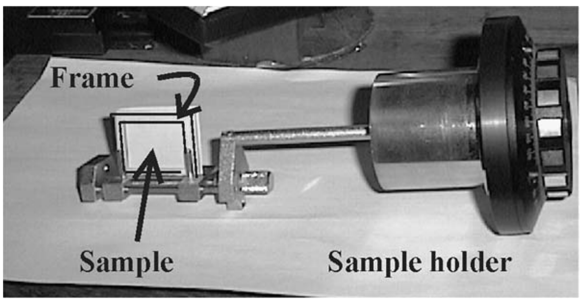

Fig. 3. Sphere's sample holder with supporting frame.

samples. Then R was calculated, as well as A ($A = 1-[R + T]$).

The value of a_T, can be determined by means of two methodologies: by exponential curve fitting to the set of values (T,h) or through direct application of Eq. (2). Knowing this value, the attenuation length, defined as

$$L = \frac{1}{a_T} \tag{4}$$

is calculated.

The former parameters also allow another important characteristic of the thermoplastic to be obtained: the complex refractive index. In an absorbing medium, the refractive index appears in a complex form, having real and imaginary parts:

$$n = n_R - i \cdot n_I \tag{5}$$

Knowing the value of a_T, it is then possible to obtain the imaginary part, n_I, related to the magnitude of the energy absorbed within the sample, using [19]

$$a_T = \frac{4 \cdot \pi \cdot n_I}{\lambda} \tag{6}$$

which can take the form

$$n_I = \frac{\lambda \cdot a_T}{4 \cdot \pi} \tag{7}$$

The real part, n_R, can be obtained through the Fresnel expression [19]

$$R = \left| \frac{n-1}{n+1} \right|^2 = \frac{(n_R-1)^2 + n_I^2}{(n_R+1)^2 + n_I^2} \tag{8}$$

where R represents the reflectance for a normal incidence on the surface. Then

$$n_R = \frac{-2 \cdot \bar{r} + \sqrt{(2 \cdot \bar{r})^2 - 4 \cdot (1 + n_I)}}{2} \tag{9}$$

with

$$\bar{r} = \frac{R+1}{R-1}.$$

This methodology was tested and applied to the analysis of high and low-density transparent polyethylene (HD and LD PE), transparent polypropylene (PP) and white high-density polyethylene films. Despite the stated objective of optical characterization for thicknesses lower than 100 μm, analysis also involved samples with thickness up to 800 μm. The laser used in the procedures was a CO_2 laser and its power was set so that no vaporization or melting occurred.

3. Results and analysis

Graphs in Fig. 4 show the variation of (a) transmittance, T, and (b) absorptance, A, with sample thickness. Standard deviation of the measurements, not plotted in these graphs, is about ±1% for T values and ±2% for A. The reflectance R is approximately constant for the different thicknesses considered, being the values experimentally obtained of 4% for transparent HD PE, 5% for transparent LD PE, 4% for transparent PP and 4% for white HD PE. Respective standard deviations are: 1.2%, 1.0%, 1.0% and 0.5%.

As previously mentioned, a_T can be determined through the application of expressions (1) or (2). In the first method (curve-fitting method), and since I/I_0 (or P/P_0) represents the transmission T, it is possible to fit a curve of the type

$$T[\%] = 100 \cdot e^{-a_T h}$$

to the experimental data.

Then, from Fig. 4(a), the relation $T(h)$ (result of curve fitting of Beer–Lambert's law to experimental points)

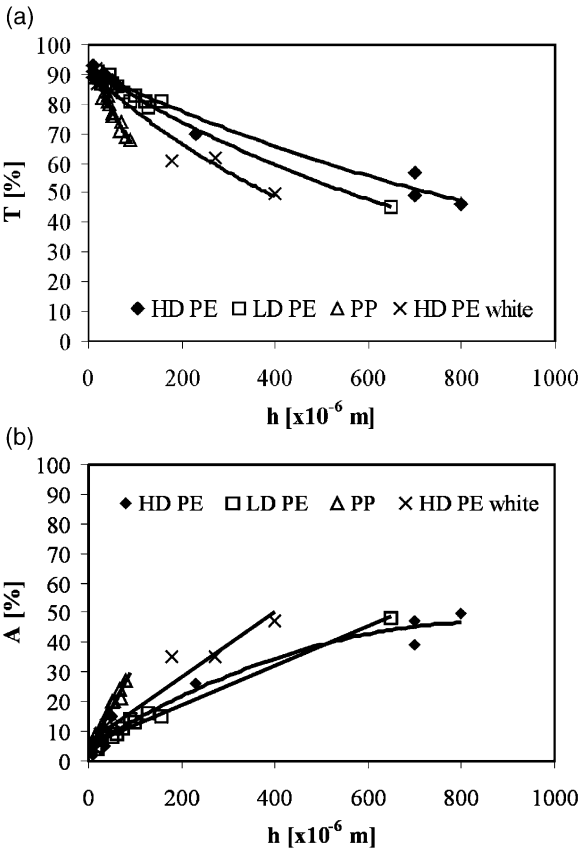

Fig. 4. Thermoplastics' (a) transmission and (b) absorption values for the thicknesses considered.

Table 1
$T(h)$ functions, obtained by curve fitting

Thermoplastic	$T(h)$
HD PE (transparent)	$T = 92 \cdot e^{-0.0008 \cdot h}$
LD PE (transparent)	$T = 92 \cdot e^{-0.0011 \cdot h}$
PP (transparent)	$T = 94 \cdot e^{-0.0038 \cdot h}$
HD PE (white)	$T = 91 \cdot e^{-0.0016 \cdot h}$

Table 2
Attenuation coefficients

Thermoplastic	a_T ($\times 10^3$ m^{-1})	$\pm\sigma_{aT}$ ($\times 10^3$ m^{-1})
HD PE (transparent)	0.82	0.04
LD PE (transparent)	1.09	0.03
PP (transparent)	3.8	0.3
HD PE (white)	1.6	0.1

can be obtained. Table 1 shows these results and Table 2 summarizes the values of a_T and their uncertainty, σ_{aT}, obtained through this methodology.

In a similar approach, Eq. (2) can be applied to previously acquired experimental data (Beer–Lambert law equation method). Fig. 5 plots the result of this procedure. Analysis of these results shows no consistency with the fact that a_T is a constant, characteristic of the material, presenting a large variation with the sample's thickness. This behavior is due to the fact that the standard deviation, σ_{aT}, associated with the propagation of experimental errors when applying relation (2), become so high for lower thicknesses that the obtained data are not reliable. This phenomenon can be seen in Fig. 6.

Nevertheless, expression (2) can be applied to determining a_T in the cases where the limit as $h \rightarrow \infty$ is achieved. Based on this hypothesis, from curves

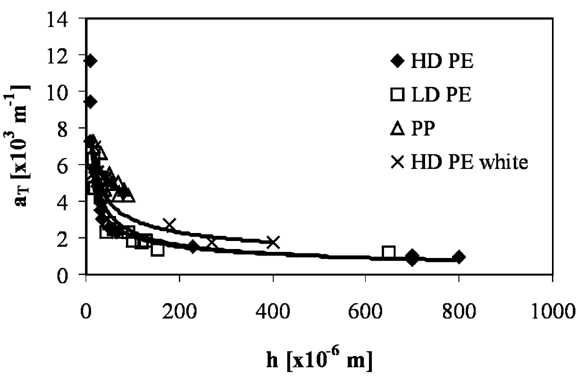

Fig. 5. Attenuation coefficient values calculated through application of Beer–Lambert law equation.

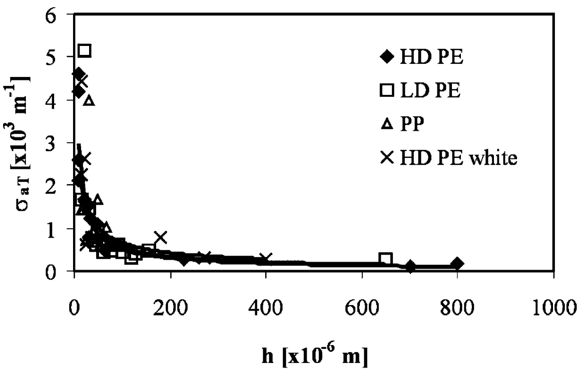

Fig. 6. Standard deviation associated with attenuation coefficients values, due to the propagation of experimental errors when applying the Beer–Lambert equation.

presented in Fig. 5 and thermoplastics available with higher thickness, one obtains, as $h \rightarrow \infty$, 1.0 mm^{-1} (±0.2 mm^{-1}) for transparent and 1.2 mm^{-1} (±0.3 mm^{-1}) for white HD PE. These values are, for the associated uncertainty, coincident with those obtained by the curve fitting method.

With the values of a_T shown in Table 2, and applying (4) the attenuation length was calculated for each thermoplastic type. Table 3 summarizes these results, as well as the associated uncertainty, σ_L. Since this parameter is defined as the depth at which the irradiance has dropped to $1/e$ (37%) of its value incident on the surface, and can be thought as a measure of radiation penetration depth, it is confirmed that the radiation is never totally attenuated for thicknesses lower than about 200 μm. Also, for thickness values lower than 100 μm, L is higher than twice the thickness of the sample (which is an important characteristic for laser processing two or more superposed thermoplastic films [10,11,20]).

From data in Table 2, the imaginary term of the material complex refractive index was also calculated in accordance with expression (7). In a similar approach, the real term was obtained by applying expression (9). Table 4 shows these values, as well as the related standard deviations.

Table 3
Attenuation lengths

Thermoplastic	L (x10^{-3} m)	$\pm\sigma_L$ (x10^{-3} m)
HD PE (transparent)	1.22	0.06
LD PE (transparent)	0.92	0.03
PP (transparent)	0.26	0.02
HD PE (white)	0.64	0.05

Table 4
Complex refractive indexes

Thermoplastic	Real part		Imaginary part	
	n_R	$\pm\sigma_{nR}$	n_I	$\pm\sigma_{nI}$
HD PE (transparent)	1.50	0.09	7.0×10^{-4}	0.3×10^{-4}
LD PE (transparent)	1.58	0.07	9.0×10^{-4}	0.3×10^{-4}
PP (transparent)	1.50	0.04	3.0×10^{-3}	0.2×10^{-3}
HD PE (white)	1.50	0.08	1.0×10^{-3}	0.1×10^{-3}

4. Conclusions

Parameters such as the attenuation coefficient, transmittance and complex refractive index have an important role in understanding the physical mechanisms of laser radiation interaction with materials. As different thermoplastic materials show different optical properties, and these properties change with the radiation applied, proper methodologies are needed, so further advances in laser processing of thermoplastics can be achieved. Since low thickness polyolefins, like polyethylene and polypropylene, have a broad application in the packaging industry, and there is great interest in the application of laser technology to their processing, the development of methodologies that can precisely characterize these polymers is a worthwhile task.

CO_2 lasers present many advantages in polymer processing, mainly based on the high absorptance that materials have to 10.6 μm wavelength. However, as thickness decreases this is no longer a valid statement since the attenuation length is larger than the sample thickness. In fact, the methods applied to the determination of the transmission and absorptance showed that for thicknesses lower than 200 μm polyethylene samples have a high degree of transparency to that wavelength. Polypropylene samples demonstrated similar characteristic for thicknesses lower than 100 μm. This transparency is characterized by absorptances lower than 20% and transmittances higher than 80%. Standard deviations between 1 and 2% are acceptable for these values.

The attenuation coefficients were obtained from transmittance curves, following a curve fitting methodology. This method has proven to reduce the influence of experimental uncertainty propagation when applying the equation from the Beer–Lambert law. However, it implies testing a broader band of thicknesses, extending the range to larger values.

The Beer–Lambert law equation method, despite being affected by experimental error propagation, can also be used if the limit $h \to \infty$ is applied to the resulting function $a_T(h)$. However, this methodology is less precise than the curve fitting method.

The equivalent attenuation length values confirmed that samples are transparent to the applied radiation: the values are higher than the samples' thicknesses.

With these parameters, the complex refractive index was calculated. Its real part prevails over the imaginary part, and could be determined to the second decimal place. This is not a very precise measurement, but when studying CO_2 laser radiation interaction with thermoplastic materials, it can be considered acceptable.

Hence, the applicability of these methodologies to the optical characterization of thermoplastic films for 10.6 μm radiation was demonstrated, and allowed data to be obtained with acceptable precision (from the laser processing point of view) with a simple and practical apparatus.

Also, it has been demonstrated that the attenuation coefficients can be determined through the application of the Beer–Lambert law, and that its related errors (appearing for lower thicknesses) are reduced through the application of a curve fitting methodology.

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