Article type: Full Paper

Light scattering by structurally anisotropic media: A benchmark with transparent wood

Elena Vasileva, Hui Chen, Yuanyuan Li, Ilya Sychugov, Max Yan, Lars Berglund, and Sergei Popov.*

E. Vasileva

Department of Applied Physics, KTH - Royal Institute of Technology, Stockholm, Isafjordsgatan 22, 16440, Sweden

E-mail: vasileva@kth.se

H. Chen

Wallenberg Wood Science Center, Department of Fiber and Polymer Technology, KTH - Royal Institute of Technology, Teknikringen 56, 10044, Stockholm, Sweden

Dr. Y. Li

Wallenberg Wood Science Center, Department of Fiber and Polymer Technology, KTH - Royal Institute of Technology, Teknikringen 56, 10044, Stockholm, Sweden

Assoc. Prof. I. Sychugov

Department of Applied Physics, KTH - Royal Institute of Technology, Stockholm, Isafjordsgatan 22, 16440, Sweden

Dr. M. Yan

Department of Applied Physics, KTH - Royal Institute of Technology, Stockholm, Isafjordsgatan 22, 16440, Sweden

Prof. L. Berglund

Wallenberg Wood Science Center, Department of Fiber and Polymer Technology, KTH - Royal Institute of Technology, Teknikringen 56, 10044, Stockholm, Sweden

Prof. S. Popov

Department of Applied Physics, KTH - Royal Institute of Technology, Stockholm, Isafjordsgatan 22, 16440, Sweden

Abstract

Transparent wood is a bio-composite material with hierarchical structure which exhibits high optical transmittance and anisotropic light scattering. Here we experimentally study the relation between anisotropic scattering and internal structure of transparent wood, and show the dependence of scattering anisotropy on material thickness, which characterises the fraction of ballistic photons in the propagating light. We discuss the limitations of the

conventional haze as it is implemented to isotropic materials, and define a modified characteristic parameter of light scattering - degree of anisotropic scattering. This parameter together with the transport mean free path (TMFP) value is more practical and convenient for characterization of the material scattering properties. We believe that generic routine described in this paper can be applied for scattering characterization and comparison of other TW materials of either different thickness, optical quality or based on various wood species.

Keywords: transparent wood; hierarchical structure; biocomposites; nanocellulose; anisotropic scattering.

1. Introduction

Transparent wood (TW) is a composite material produced from natural wood, and exhibits improved mechanical properties ^[1, 2] with added optical functionalities. It was initially demonstrated by Fink in 1992 ^[3] for investigation of internal structure of wood species. However during recent years, the interest towards TW was significantly renewed in several research areas: material science designing multifunctional wood composites ^[2, 4-6], energy saving technology focused on application of TW in solar cells ^[7], and photonics dealing with light sources based on TW with additives ^[8, 9].

Natural wood consists of three main components which have different refractive indices: cellulose (1.525 with light propagating perpendicularly to the cellulose fibers, and 1.596 along with the fibers), hemicellulose (1.532), and lignin (1.61) ^[3]. While holocellulose (cellulose and hemicellulose) is optically colorless, lignin is the main source of absorption (about 80-95% of wood light absorption) in the visible range ^[10]. Thus, lignin removing ^[11] or deactivating the chromophores inside lignin is required to make wood transparent ^[12]. Although major part of lignin is removed during chemical modification, some residual amount (1-3%) still remains in the wood ^[11] introducing minor absorption. A delignified wood (**Figure 1a**) is not yet transparent due to strong scattering of light, which occurs on the interfaces between the material structural components and air-filled voids between/within them. To minimize such scattering, the delignified template is infiltrated with a polymer of refractive index approximately matching the wood compounds. In several previous studies, PMMA whose refractive index is close to the average refractive index of delignified wood

template (≈ 1.53)^[11] was implemented. However, there always exists minor refractive index mismatch between the wood components and infiltrated polymer that leads to the light scattering. Moreover, as a consequence of mesoporous hierarchical structure of a wood template^[13] with feature sizes scaling from micrometers - cellulose fibers, vessels, rays^[14] (schematically presented in **Figure 1b**) down to nanometers (nanofibrils),^[1, 15] polymer infiltration process is not perfect, e.g. due to surface compatibility issues. This leads to formation of structural air-filled voids in the walls of the cellulose fibers consisting of nanofibrils (**Figure 1c**). It should be noted that additional air-filled voids can be also created via polymer shrinkage during polymerization^[16] (**Figure 1c**). Hence, scattering in TW is caused by several factors: minor mismatch of the refractive indices between the delignified wood template and infiltrated polymer, multi-scale porous hierarchical structure of the wood consisting of macro-, meso and nano-pores, and presence of air-filled voids. Thus, despite relatively high optical transmittance, TW is, in fact, an optically anisotropic scattering medium.

Traditionally optical properties of TW have been characterized *via* measuring the light total hemispherical luminous transmittance (referred as transmittance in the literature about TW) and haze^[11, 17]. While transmittance describes optical losses of a material, i.e. the fraction of the optical energy flux passing through the material, haze is important to characterize scattering properties of medium in terms of image content^[19]. In other words, haze represents dissolution of a picture seen through a material. It can be measured as the loss of contrast due to deviation of incident optical beam from rectilinear propagation through the medium caused by light scattering. In previous studies, the conventional technique for measuring transmittance and haze in plastics (American Society for Testing and Materials ASTM D1003-13 for plastics^[19]) has been applied to TW. According to the ASTM standard, the transmitted light is collected with an integrating sphere, so total hemispherical luminous transmittance (referred as transmittance in the literature about TW), i.e. integrated transmission, is determined. One needs to notice that its value is higher than a regular luminous transmission which is measured by placing a specimen at some distance from the entrance port of the integrating sphere^[19]. Haze characterization has utilized the same standard as the procedure described in the reference^[19]. However, the ASTM haze measurement is applicable for materials, which scatter light homogeneously and uniformly.

On the other hand, TW is highly anisotropic substance due to its oriented fibrous structure; correspondingly, a TW sample will scatter light differently along two transverse directions depending on light propagation direction. Until recent, very few research reports mentioned anisotropic transmitted intensity distribution of the beam propagating through the TW material ^[9, 17, 18, 20]. However, in those reports authors analyse either light guiding by fibers inside the transparent wood ^[9, 18] or briefly mentioned anisotropic optical (transmitted light distribution) and mechanical properties ^[17]. Here we analyze, both qualitatively and quantitatively, anisotropic scattering of light propagating perpendicularly to the direction of fibers.

Considering the reasoning mentioned, the conventional ASTM haze measurement procedure cannot accurately characterize highly scattering anisotropic material as TW. Thus, special care should be taken while applying this parameter for description of scattering properties of the material. Therefore, there is a need in developing the generic routine for characterization of scattering in this class of structures, which include bio-based, organic, and various synthetic materials.

In this paper we discuss experimentally observed light transmission and anisotropic scattering in transparent wood which are associated with its internal hierarchical structure. In order to characterize the anisotropic scattering, we developed the generic routine and suggested the term “degree of anisotropic scattering” instead of commonly used “haze” (applicable for the analysis of isotropic scattering media). We have also discussed the limitations of the haze method for TW characterization. Considering the above and following arguments in the paper, we think the introduced parameter is more efficient and informative to characterize anisotropic scattering properties of TW and similar media. Thus, we believe besides the experimental investigation of scattering in TW, the suggested approach can be also used for comparison of TW samples of different optical quality, thickness, and/or wood species.

2. Experimental section

2.1. Fabrication of transparent wood samples

In our study, acetylated TW was prepared in four main steps based on our previous work ^[16]: balsa wood template (density: 160 kg/m³, purchased from Wentzels Co. Ltd, Sweden) cut from tangential section of the tree was delignified using sodium chlorite

(NaClO₂, Sigma-Aldrich) to remove lignin. Then the sample was acetylated by acetate anhydride (Sigma-Aldrich) to reduce the interfacial gaps between the polymer and cell wall in the TW through improving the surface compatibility between wood template and polymer after acetylation. After that, the acetylated balsa was delignified again to remove the color from the chemical and finally infiltrated with pre-polymerized methyl methacrylate (PMMA) to form TW.

To measure the angular distribution of scattered light we prepared the set the samples of different thicknesses. For this purpose, a sample of the initial thickness of 7.7 mm was gradually cut to the desired thickness and carefully polished at each measurement step, so overall, we have had a set of 15 thicknesses for the investigation. We used the same piece of TW to exclude any possible differences of the wood structure on the results since, for example, latewood and earlywood of the same wood species have different sizes of structural features ^[13]. The samples were polished with a polishing machine (Buehler). Each polishing consists of several stages, for which different polishing disks of gradually decreasing grain size were used to ensure the optical quality and exclude light scattering from the sample facets in visible. The fine polishing was done with where minimum grain size of the polishing disk was equal to 3 μm. All samples were polished till optical quality in exactly the same manner; therefore we ensured the polishing process and related surface roughness might have only negligible effect (if any) on the scattering distribution.

2.2. Scattering property characterization

Experimental setup for measuring the scattering properties of TW is schematically presented in **Figure 2**. It includes a He-Ne laser (633 nm) with a beam (diameter of 1.5 mm) passing through a sample (TW) and a detector/power meter (D) placed on a rotation stage with the radius of 13.6 cm. The laser was circularly polarized by means of a linear polarizer (LP) and a properly oriented quarter wave plate (QWP) for the visible spectral range of light. The input aperture of the power meter was limited to 1.8 mm, which roughly corresponds to the scattering angle of 1°. The convolution of measurements between neighbor points was avoided since an angular distribution has been measured with the step of 3°. The scanning of intensity distribution was in a horizontal plane associated with x-axis on the **Figure 2**. Blue bar-chart there schematically represents the angular distribution of scattering intensity.

In order to measure the scattering intensity distribution in two orthogonal directions, the TW sample was rotated in a way that wood fibers (and vessels as visible reference in **Figure 1**) are oriented to the plane of measurement (angular scanning of the detector) either parallel or perpendicularly. The photos of the sample orientations with corresponding intensity distribution are shown in the inset of **Figure 2**. By precise alignment of the sample holder, we ensured the incident beam always hit the same spot on the sample while it was rotated. In more details, the measurements were performed according to the following routine: first, the sample was located in such a way that wood fibers were oriented in the plane of angular scanning; at the second step, the sample was rotated by 90° , so the fibers were in vertical position. Since the incident laser beam is rotationally symmetric (and circularly polarized), such rotation of the sample was equivalent to the angular detector scanning in the transverse direction. As a result, the angular intensity distribution was effectively collected in two coordinate planes (**Figure 2**). The angular scanning was repeated twice for both horizontal and vertical sample orientations for each sample thickness, and averaged results were analyzed.

3. Results and discussion

The TW anisotropy responsible for light scattering is related to an inherent geometry of main structural components in micrometer size scale: vessels, fibers, and rays (**Figure 1**). The main contribution is, however, made by wood fibers, the major structural component of TW. Fibers and vessels are aligned in same direction, while rays are perpendicular to them (**Figure 1**). Vessels are of relatively large size (typical diameter $220\text{ }\mu\text{m}$ for balsa) ^[14] and visible by a naked eye (inset **Figure 2**), so they were used as orientation reference (horizontal or vertical) for the sample's position in the setup. It is known that in case of dielectrics (refractive index in range 1-3) scattering is the most efficient when the sizes of scattering objects are comparable to the light wavelength. Thus, one can conclude that most efficient scattering takes place on the walls (up to $3\text{ }\mu\text{m}$ thickness) of wood fibers (typical diameter of $30\text{ }\mu\text{m}$ in balsa) consisting of nanofibrils (several nm) and at the interfaces between the solid material and air-voids discussed above ^[14].

The measurements demonstrate high anisotropy of light scattering by TW material in two perpendicular planes relative to the fiber orientation. Several examples of the distribution

for different material thicknesses are displayed in **Figure 3**. It is easy to see, that the light scattering is considerably stronger in the plane perpendicular to the orientation of fibers (and vessels) inside TW material (marked in pink color in the angular histogram). In contrast, the distribution in the plane parallel to the TW preferred direction is narrower. Referring to the wood structure (**Figure 1**), the sample can be treated as a superposition of two cross-oriented volume “diffractive gratings” with discrete refractive index variation: one vertical (according to layout in **Figure 1**) consisting mainly of fibers (vessels do not contribute significantly due to large diameter and relatively low volume fraction) and the other, in perpendicular orientation, consisting of rays. Here, we refer to particular wood features such as rays, vessels, and fibers for the simplicity of narration. However, one needs to keep in mind that scattering is initiated not only by the features themselves but rather inside or in vicinity of their walls where refractive index mismatch takes place - between air-filled voids and infiltrated polymer as well as wood substances with slightly different refractive index (discussed above). Since the volume fraction of rays is much smaller than of fibers, the scattering by rays is weaker which is reflected in narrower angular diagram (**Figure 3** blue bar-chart). The “gratings” are far from ideal regular structures that result in smoothened intensity distributions on angular diagrams, rather than exhibiting clear peaks of diffractive orders typical for conventional diffractive gratings.

In order to analyze light scattering via beam divergence in a way similar to angular radiation diagrams, we consider the full-width half maximum (FWHM) of the angular transmitted intensity distribution. Its dependence on the sample thickness is presented in **Figure 4** for vertical and horizontal orientations of the wood samples. The dependence has two noticeable regions. The first one associated with the thickness less than 2 mm where FWHM values for both orientations are nearly constant. For the thicker samples, the output beam directionality degrades significantly. In particular, light beam expands 2.5 times faster in the plane perpendicular to the fiber orientation (pink dots) than it does in the plane parallel to the fibers (blue dots). Nevertheless, both curves are linear, thus, light is scattered with constant rates determined by curve slopes (3.24 for horizontal vs 8.11 for vertical positions). It is reasonable to assume that the existence of these two regions associated with ballistic photons and the transport mean free path (TMFP). TMFP is established term in the theory of optical scattering that is defined as the distance after which the photon direction of propagation is fully randomized and is not related to the initial direction; in other words, the ballistic

photons are absent. Thus, TMFP value is around 2 mm for the balsa wood tested which is in good agreement with the one for non-ordered organic tissues (around 1 mm) ^[21]. The particular value of TMFP is not an absolute constant, but rather is a characteristic value related with the optical scattering efficiency established by wood structure. Therefore, it depends on the wood morphology varying for different wood types (early- and late-wood) and species (ash, birch, pine). Although the value of TMFP for the same sample might differ, it still remains the same within experimental inaccuracy, as the internal structural features do not vary significantly.

One can come to the similar conclusion about TMFP value for the material under investigation by analyzing the data in **Figure 5**, which demonstrates the transition from a sharp image behind the TW sample to more blurred one. Thus, for the samples thinner than TMFP value most of the photons are still ballistic (sharper images on the left side), while for the thicker samples ballistic photons are almost absent (blurred image with no features resolved on the right side).

A simplified estimation of the TMFP parameter can be performed with a direct transmittance measurement within the narrow solid angle about 1° - 1.5° . The direct transmission measurement results, normalized to the maximum intensity for the thinnest sample, are shown in **Figure 6**. Again, one can see an abrupt drop of intensity dependence for the sample thickness around 2 mm (attributed to TMFP value) followed by the decrease of the forward transmitted intensity with much slower rate. This can be explained by the lack of ballistic photons at the output and the dominance of multiply scattered photons from the material volume.

According to the standard ASTM procedure for conventional (isotropic) materials with scattering properties, an integrating sphere is utilized for haze measurements ^[19]. Haze is defined as being proportional to the ratio of diffused photons to the total amount of transmitted photons, with corrections accounting for sphere imperfections (scattering by instrument) and light backscattering from a sample. The standard setup should meet certain geometric requirements: the input and output ports of the sphere should be located at least 170° of arc between their centers, and their total areas do not exceed 4% from the internal reflecting area of the sphere, light trap port should subtend 8° angle at the center of input port along the propagating beam ^[19]. As discussed above, non-uniform anisotropic intensity distribution beyond a light cone of 8° (**Figure 3**) would create extra limitations for

applicability of ASTM procedure for characterization of TW material. By the definition given in ASTM ^[19], haze is the percentage of transmitted light that is scattered beyond a specified (2.5°) angle from the direction of propagation. By physical meaning, this is related to the contrast reduction of an image viewed through the material. It does not refer to any anisotropy in the distribution as it is assumed to be a uniform one. According to our measurements, haze definition might be applied for TW with some restrictions: the deviation angle should be restricted to 1° - 1.5° where the angular distributions for two sample orientations are still comparable (**Figure 3**) and so the values of direct transmittance for them are equal (**Figure 6**). On the other hand, the material thickness should not exceed the value where the FWHM values start to deviate noticeably, i.e. about 2 mm (**Figure 4**). Thus, conventional haze analysis can be applied to anisotropic materials with sample thicknesses below TMFP and with smaller detector aperture corresponding to 1° - 1.5° divergence angle.

For more quantitative estimation of the scattering anisotropy, it is instructive to introduce a measure of the *degree of anisotropic scattering* defined as a ratio of the FWHM for horizontal (respectively vertical) angular intensity distribution to the sum of FWHMs of vertical and horizontal distributions. Dependence of this value from the sample thickness is shown in **Figure 7**. The physical meaning of the introduced value can be understood as follows. In the case of a uniform light distribution, the values of the degrees of anisotropic scattering are the same and equal to 0.5. In an opposite case of highly anisotropic distribution, the value of the degree of anisotropic scattering tends to unity for the preferred scattering direction (either vertical or horizontal) and to zero for the other one. In our case, scattering is anisotropic with the favorable direction perpendicular to wood fibers (pink dots **Figure 7**). The uncertainties in the measurements are originated from different position of the incident beam on a sample surface (the error bars on the **Figure 6 – 7**). By changing the position of the beam over the sample, different combinations of structural features illuminated, thus scattering (efficiency and TMFP values) are statistically affected.

The degrees of anisotropic scattering for both horizontal and vertical distributions are nearly constant and independent on the sample thickness. That means that there are no changes in the sample structure (material anisotropy). This result agrees with data in **Figure 4**, where FWHM is nearly linearly dependent from the sample thickness for both directions. According to the introduced definition, the degree is a ratio of two functions which have constant derivatives, thus, are in linear relation to each other.

4. Summary

Summarizing, we report on the study and analysis of light scattering and transmission in optically anisotropic material – transparent wood. Anisotropy of the light scattering is attributed to structural anisotropy of the material introduced by co-aligned vessels and fibers, as well as perpendicularly oriented rays. We demonstrate experimental evidences that in order to characterize scattering properties of TW, the haze (commonly used for the analysis of isotropic scattering media) should be applied with certain restrictions: the sample thickness should be smaller than the TMFP, and the detector aperture should be considerably limited. The first constraint is attributed to the presence of ballistic photons which disappear at the distances longer than TMFP (their trajectory is totally randomized and output light is the result of multiply scattering inside the material), while the second one is caused by non-uniform spatial distribution of ballistic photons. These limitations are essential for the haze measurement according to the ASTM standard. Thus, the applicability of a conventional ASTM routine to assess material haze from isotropic scattering media requires reconsidering in this case. Therefore, instead of haze, we suggest more practical measure to describe the scattering properties of TW – the degree of anisotropic scattering, which together with the TMFP value can be useful for characterization of TW in image content with the loss of contrast (similar to haze definition). This formalism can be also applied for characterization of other materials occupying the intermediate niche between fully ordered and disordered media. From practical perspectives, anisotropic scattering can be both advantageous and disadvantageous feature of TW materials depending on particular applications, where other beneficial features of TW are strongly desirable – good thermal isolation, light weight, mechanical hardness, decreased fragility, etc.

7. Acknowledgments

Authors acknowledges support from The European Research Council, project "Wood NanoTech", gr. # 742733; Swedish Research Council, project "Electromagnetic coherence: physical effects and applications", gr. # 621-2012-4421; The Linné Center in Advanced Optics and Photonics ADOPT at KTH Royal Institute of Technology.

Received: ((will be filled in by the editorial staff))
Revised: ((will be filled in by the editorial staff))
Published online: ((will be filled in by the editorial staff))

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Figure 1. a) SEM picture of native delignified balsa wood template; b) schematic structure of the TW sample; c) SEM picture of balsa wood template filled with PMMA.

Figure 2. Schematic layout of experimental setup. LP-linear polarizer, QWP- quarter wave plate, TW-sample of transparent wood, D – detector, S-screen. Inset: photo of the sample and corresponding photo of scattered light after the sample.

Figure 3. Normalized angular intensity distribution of scattered light for samples of different thickness (d) at horizontal (blue) and vertical (pink) fibers orientation:
a) $d = 0.7\text{mm}$; b) $d = 3.6\text{ mm}$; c) $d = 5.2\text{ mm}$; d) $d = 7.7\text{mm}$.

Figure 4. Dependence of FWHM of angular intensity distribution of scattered light from the sample thickness for two orientations of a sample.

Figure 5. Photos of the samples of different thicknesses (marked at the bottom right corner) placed in the contact with the post stamp. The series of the photos demonstrates the dependence of transmittance and image visibility behind the TW piece from its thickness. Vessels/fibers orientation is marked on the sample (right top corner).

Figure 6. Dependence of normalized direct intensity from the sample thickness: a) for horizontal and b) vertical fibers orientation.

Figure 7. Dependence of the degree of anisotropic scattering from the sample thickness.