Effective structural chirality of beetle cuticle determined from transmission Mueller matrices using the Tellegen constitutive relations

Hans Arwin, Roger Magnusson, Kenneth Järrendahl, and Stefan Schoeche

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Hans Arwin,1,a) Roger Magnusson,1 Kenneth Järrendahl,1 and Stefan Schoeche2

AFFILIATIONS
1Department of Physics, Chemistry and Biology, Linköping University, Linköping SE-58183, Sweden
2J.A. Woollam Co., Inc., 645 M Street, Suite 102, Lincoln, Nebraska 68508

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a)Electronic mail: hans.arwin@liu.se

ABSTRACT

Several beetle species in the Scarabaeoidea superfamily reflect left-handed polarized light due to a circular Bragg structure in their cuticle. The right-handed polarized light is transmitted. The objective here is to evaluate cuticle chiral properties in an effective medium approach using transmission Mueller matrices assuming the cuticle to be a bianisotropic continuum. Both differential decomposition and nonlinear regression were used in the spectral range of 500–1690 nm. The former method provides the sample cumulated birefringence and dichroic optical properties and is model-free but requires a homogeneous sample. The materials chirality is deduced from the circular birefringence and circular dichroic spectra obtained. The regression method requires dispersion models for the optical functions but can also be used in more complex structures including multilayered and graded media. It delivers the material properties in terms of model functions of materials’ permittivity and chirality. The two methods show excellent agreement for the complex-valued chirality spectrum of the cuticle.

I. INTRODUCTION

Several beetle species in the Scarabaeoidea superfamily reflect left-handed polarized light due to a circular Bragg structure in their exoskeleton, also called cuticle.1 The effect is illustrated for the scarab beetle Cetonia aurata (Linnaeus, 1758) in Fig. 1, where it is seen that the beetle appears nonreflecting (black) when viewed through a right-handed polarizer and preserves its color when viewed through a left-handed polarizer. The cuticle is composed of chitin and proteins and is essentially nonabsorbing, and the right-handed polarized light is transmitted as illustrated schematically in Fig. 1. The circular Bragg structure is chiral and is also called a Bouligand structure and has been visualized in several beetles by electron microscopy.1,3–7 The optical features of such biological reflectors include structural colors, polarization, and depolarization and have been explored in numerous investigations based on spectral reflectance.7–9 A more advanced methodology, compared to reflectance measurements, is Mueller matrix ellipsometry, which has the advantage that it allows polarization and depolarization features to be quantified. Mueller matrix measurements on the chiral beetle structure were pioneered by Goldstein9 and Hodgkinson et al.10–12 Spectral reflection Mueller matrices can be recorded with high accuracy using commercial instruments and are very rich in information about complex electromagnetic structures. Calculations of polarization states including depolarization of reflected light for any incident state of polarization have been demonstrated for several beetles.11,12 By using electromagnetic modeling, structural details like layer thicknesses, pitch of Bragg structures, pitch distributions, and refractive indices of biaxial constituents can be extracted.12,13,14 A sum decomposition of a Mueller matrix provides a phenomenological description of beetle cuticle reflection in terms of optical elements like retarders and polarizers.15 Transmission Mueller matrices allow quantification of chirality in terms of structural birefringence and dichroism.16

In this work, the objective is to evaluate chiral properties of the beetle cuticle as a bianisotropic continuum in a model-free effective medium approach based on differential decomposition of
II. THEORETICAL BACKGROUND

A. Tellegen constitutive relations

The beetle cuticle studied here exhibits chirality. The origin is not molecular but is classified as structural and can be well represented with an electromagnetic model when analyzed using reflection Mueller matrices. Here, we employ an effective medium approach to quantify the effective chirality of the cuticle. The full constitutive relations are used, which in the so-called Tellegen representation (also called EH-representation) are

\[
D = \varepsilon_0 \varepsilon E + \varepsilon_0^{-1} \varepsilon \chi H, \quad (1a)
\]

\[
B = \mu_0 \mu I + \mu_0 \mu_0 \mu, \quad (1b)
\]

where \(D\), \(E\), \(H\), and \(B\) are the electric displacement field, electric field, magnetic field, and magnetic flux density, respectively, \(\varepsilon_0\) and \(\mu_0\) are the vacuum permittivity and permeability, respectively, and \(c_0\) is the speed of light. Material properties are given by the permittivity tensor \(\varepsilon\), the permeability tensor \(\mu\), and the magnetoelectric tensors \(\chi\) and \(\chi\).

With a time dependence \(e^{-i\omega t}\), it holds that \(\varepsilon = \chi + i \chi\), where \(\chi\) is the nonreciprocity tensor and \(\chi\) is the chirality tensor. In this case, the medium is reciprocal, which implies \(\chi = 0\) and \(\chi = -\chi^*\), where \(T\) indicates the transpose. We, thus, have \(\varepsilon = -\varepsilon^*\). A dielectric circular Bragg structure probed with a plane wave along its helical axis can be modeled with \(E = \text{diag}(e_{xx}, e_{yy}, e_{zz})\) and \(H = \text{diag}(k_x, k_y, k_z)\). Here, an xyz Cartesian coordinate system is used with the \(z\) axis along the helical axis. The permittivity elements are related to the complex-valued refractive indices by \(N_j = n_0 + ik_j = \sqrt{\varepsilon_j} (j = x, y, z)\), where \(n_0\) is the refractive index and \(k_j\) is the extinction coefficient. At normal incidence, there is no sensitivity to the absolute values of the refractive indices \(n_x\) and \(n_z\) in the used methodology since the absolute phase of the light beam is not accessible. However, phase differences are measured in an ellipsometric measurement, and \(\Delta n = n_y - n_x\) is accessible. As we use irradiance normalized data in the analysis, there is no sensitivity to isotropic absorption either, but the difference \(\Delta k = k_y - k_x\) is accessible. Furthermore, there are no fields from the probe beam in the \(z\)-direction and \(e_z\) can be set arbitrarily. Here, we use \(e_z = e_z\). Similarly, there is no sensitivity to \(k_z\), which also can be assumed arbitrarily. We choose to set \(k_2 = k_x\), and \(k\) can, therefore, be set to \(k I\) with the complex-valued scalar \(k = k_{\text{Re}} + ik_{\text{Im}}\) and the identity matrix \(I\). Finally, we set \(\mu = I\). The relevant constitutive relations can then be written as

\[
D = \varepsilon_0 \varepsilon E + \varepsilon_0^{-1} \varepsilon \chi H, \quad (2a)
\]

\[
B = -\varepsilon_0^{-1} i \chi E + \mu_0 \mu H, \quad (2b)
\]

or in a more compact form

\[
D = \varepsilon_0 \varepsilon \text{diag}(N_x^2, (N_x + \Delta n + i\Delta k)^2, N_y^2) E + \varepsilon_0^{-1} i \chi \text{diag}(N_z^2), \quad (3a)
\]

\[
B = -\varepsilon_0^{-1} i \chi \text{diag}(N_y^2, (N_y + \Delta n + i\Delta k)^2, N_z^2) E + \mu_0 \mu \text{diag}(N_x^2) \quad (3b)
\]

B. Differential decomposition

All birefringent and dichroic properties of a sample are directly obtained by taking the logarithm \(\mathbf{L} = \ln \mathbf{M}\) of a transmission Mueller matrix \(\mathbf{M}\) provided that the sample is homogeneous along the beam path. The matrix \(\mathbf{L}\) can be decomposed as \(\mathbf{L} = \mathbf{L}_0 + \mathbf{L}_0\), where \(\mathbf{L}_0\) carries the depolarization properties and \(\mathbf{L}_0\) is given by

\[
\mathbf{L}_0 = \begin{bmatrix}
0 & LD & LD' & CD \\
LD & 0 & CB & -LB' \\
LD' & -CB & 0 & LB \\
CD & LB' & -LB & 0
\end{bmatrix}, \quad (4)
\]

where linear (\(L\)) and circular (\(C\)) birefringence (\(B\)) and dichroism (\(D\)) are defined by \(LD = 2\pi d(k_x - k_y)/\lambda\), \(LB = 2\pi d(n_y - n_x)/\lambda\), \(LD' = 2\pi d(k_x + k_y)/\lambda\), \(LB' = 2\pi d(n_x + n_y)/\lambda\), \(CD = 2\pi d(k_x - k_y)/\lambda\), and \(CB = 2\pi d(n_x - n_y)/\lambda\). Here, \(\lambda\) is the wavelength, \(d\) is the sample thickness, and subscripts indicate polarizations with
directions along \((x/y)\) and \(±45°\) from the reference coordinate system and for left and right \((l/r)\) circular polarizations. Notice that the elements of \(L_m\) are cumulated values along the beam path in the sample and, thus, are considered as sample properties as they depend on sample thickness. \(LB, LB_0,\) and \(CB\) are given in units of radians, whereas \(LD, LD_0,\) and \(CD\) are dimensionless. Different conventions are used for signs in \(L_m\) and in the definition of its elements. Here, we use \(L_m\) conventions as in Ref. 19 and conventions for its elements as above.

The relation between \(κ\) and \(CB\) and \(CD\) is given by

\[
κ = κ_{Re} + iκ_{Im} = -\frac{λ}{4πd}(CB + iCD),
\]

which relates the effective materials parameter \(κ\) and the phenomenological sample parameters \(CB\) and \(CD\). Equation (5) follows if \(e^{-iωt}\) convention is used and if \(κ\) enters the wave vector \(q\), where \(q = 2π/λ(\sqrt{εμ_p + κ}/C_0)\), with \((+)(−)\) representing right(left)-handed polarization.

III. EXPERIMENT

A. Sample preparation

The specimen of the beetle \(C. aurata\) was collected locally. The outermost layer of the cuticle is a thin epicuticle consisting mainly of wax2 and with a thickness around 500 nm in \(C. aurata\).13 Below the epicuticle is the exocuticle that holds the helicoidal structure providing chirality, and that is the main structural element responsible for color and polarization features. Farthest in the cuticle, there is a thick and relatively soft endocuticle. The ellipsometric transmission measurements were performed on one of the elytra (cover wings) that was removed from a beetle and carefully scraped manually with a sharp knife on the inside to remove the endocuticle. In this way, the exocuticle could be exposed in an area of a few square millimeters. The epicuticle, which was left intact, has been found to be uniaxial12 with its optic axis perpendicular to the surface plane, and thus, it will not affect polarization in normal incidence transmission measurements.

B. Mueller matrix ellipsometry

Transmission Mueller matrices were measured in the spectral range of 300–1690 nm at normal incidence with a dual rotating compensator ellipsometer from J. A. Woollam Co., Inc. However, for \(λ < 500\) nm, the transmittance is too low for determination of a Mueller matrix of sufficient quality, and analysis was restricted to \(λ > 500\) nm. A Mueller matrix is a \(4 × 4\) matrix providing a complete description of polarizing and depolarizing properties of a sample for light with any input polarization described by a so-called Stokes vector. The Mueller matrix elements \(M_{ij}\) \((i, j = 1…4)\) are normalized to the total reflectance, i.e., element \(M_{11}\), according to \(m_j = M_{ij}/M_{11}\). Further details about the Mueller–Stokes formalism can be found elsewhere.20

With focusing optics, the spot size was reduced to approximately 50 μm. For lenses with 28 mm focal length, the beam divergence is less than \(±3.8°\), which results in small \(z\)-components from the incident transverse field. The effect of this small component can be neglected. Measurements were performed on the elytron from a \(C. aurata\) beetle with results as shown in Fig. 2. The sample azimuth \(φ\) with respect to the instrument \(xy\) frame of reference is unknown and fitted in the nonlinear regression. Optical activity is

![FIG. 2. Normalized transmission Mueller matrix measured on an elytron from a specimen of the scarab beetle Cetonia aurata.](image)
observed in the antidiagonal with a resonance around a wavelength of \( \lambda = 560 \) nm. However, the noise level is large for \( \lambda < 500 \) nm due to low transmittance, and modeling is restricted to \( \lambda > 500 \) nm.

Nonlinear regression of the data in Fig. 2 was performed with the WVASE software (J. A. Woollam Co., Inc.) using the Levenberg–Marquardt method.\(^{21}\) The results of a fit are values of the best-fit parameters and their 90\% confidence limits. A structural model with a single homogenous layer with thickness \( d = 20 \) \( \mu \)m was assumed. In normal incidence transmission studies, optical properties perpendicular to the sample surface are not accessible as discussed above. The same holds for in-plane optical functions, and only their differences, i.e., birefringence and dichroism, are modeled in terms of \( \Delta n = n_x - n_y \) and \( \Delta k = k_y - k_x \), respectively. In the analysis, Eq. (3) was used with Gaussian dispersion models for \( \kappa = \kappa_{1m} + i \kappa_{2m} \), which is given by

\[
\kappa_{1m}(E) = \sum_{j=1}^{2} \left[ A_j e^{-\frac{(E-E_j)^2}{\Gamma_j}} - A_j e^{-\frac{(E+E_j)^2}{\Gamma_j}} \right],
\]

\[
\kappa_{2m}(E) = \kappa_0 + KK(\kappa_{1m}),
\]

where \( A_j, E_j, \) and \( \Gamma_j \) are amplitude, resonance energy, and broadening of resonance \( j \), respectively; \( \kappa_0 \) is a constant; and KK stands for the Kramers–Kronig transform. Notice that the spectral dependencies here are written versus photon energy \( E = \hbar c/\lambda \) (in units of eV), where \( h \) is Planck’s constant. The linear birefringence \( \Delta n \) and linear dichroism \( \Delta k \) are modeled with a Cauchy dispersion and an Urbach tail, respectively,

\[
\Delta n(\lambda) = A + B \lambda^2 + C/\lambda^4,
\]

\[
\Delta k(\lambda) = A_U e^{B_U(1/\lambda^4)},
\]

where \( A, B, C, A_U, \) and \( B_U \) are fitting parameters. The band edge \( C_U \) is not fitted as it correlates with \( A_U \).

IV. RESULTS AND DISCUSSION

A. Nonlinear regression

Figure 3 shows the measured Mueller matrix from Fig. 2 in the spectral range used for nonlinear regression. Below 500 nm, the transmission is very low and systematic errors affect the fitting. Above 800 nm, no further information is found, and the data are omitted for clarity. The best-fit model-calculated data are also shown in Fig. 3. The best-fit parameters for the two Gaussian resonances are found in Table I. Moreover \( \kappa_0 = -9.2 \times 10^{-5} \pm 2 \times 10^{-6} \). The Cauchy and Urbach parameters in Eq. (7) are \( A = 0.0012 \pm 0.0001, B = -0.00017 \pm 0.00002 \mu m^{-4}, C = -0.000028 \pm 0.000004 \mu m^4, A_U = 0.00086 \pm 0.00003, \) and \( B_U = -0.00011 \pm 0.00005 \mu m, \) and the fitted sample azimuth is \( \phi = 128 \pm 1^\circ \).

For elements \( m_{41} \) and \( m_{14} \), the fits are excellent. In general, the fits for the diagonals are good except for in the range of 500–550 nm for some elements. For the remaining elements, the fits show various degrees of deviation from the data, especially for shorter wavelengths and close to the Bragg resonance.

![FIG. 3. Experimental Mueller matrix measured (blue solid curve) on Cetonia aurata and best fit (red dashed curve) obtained by nonlinear regression.](avscitation.org/journal/jvb)
B. Differential decomposition

Figure 4 shows the parameters in the matrix $L_m$ calculated from the data in Fig. 2. The dominating features are the $CB$ and $CD$ elements. The circular Bragg resonance is observed with a peak in $CD$ at 571 nm. The $CD$ spectrum has a shoulder on the long-wavelength side, indicating that the structure is more complex than a single helicoidal structure. The $CB$ spectrum is Kramers–Kronig related to the $CD$ spectrum, and together $CB$ and $CD$ are referred to as the Cotton effect. The $CD$ shows a positive background level as seen for short wavelengths and is referred to as a positive Cotton effect.

The linear birefringence spectra ($LB$, $LB'$) and dichroism spectra ($LD$, $LD'$) are one order of magnitude smaller than $CB$ and $CD$. $LB$ and $LD$ are close to zero, and both $LB'$ and $LD'$ have small but nonzero values indicating that the sample is in an orientation at which the optical axis is oriented at ±45°. This is consistent with the regression result $\phi = 128°$, which due to 180° symmetry is equivalent to −52°, i.e., 7° from −45°. The four elements $LB$, $LD$, $LB'$, and $LD'$ are nearly nondispersive except for features in the spectral range for the Bragg resonance.

C. Comparison between differential decomposition and nonlinear regression analysis

Figure 5 shows $\kappa = \kappa_{Re} + i\kappa_{Im}$ in Eq. (6) according to the best-fit model data in Fig. 3 compared with $\kappa = \kappa_{Re} + i\kappa_{Im}$ calculated from $CB$ and $CD$ from Fig. 4 using Eq. (5).

The agreement between the two methods is very good with some deviations in $\kappa_{Re}$ at short wavelengths. We attribute this to systematic errors due to low sample transmission for $\lambda < 550$ nm. The shoulder on the long-wavelength side of the Bragg resonance is well reproduced by the double resonance dispersion model used in the regression.

D. Discussion

The chirality spectra determined with the two methods are very close. The differential decomposition is only a transformation of primary data and all information is preserved, which is very advantageous as all details can be observed. In the present case, the cuticle is not homogeneous along the beam path, which implies that the cuticle effective anisotropic properties are obtained rather than the intrinsic properties of the cuticle materials. This is not a drawback as we compare with nonlinear regression in which a homogeneous effective medium is assumed. However, this is not a requirement in the regression approach, and more complex structures can be analyzed including graded or multilayered structures. For example, chirality of a buried layer may be extracted. The drawback with regression analysis is that if the model used is wrong or incomplete, false results may be obtained.

It may be of interest to determine the maximum specific rotation of the cuticle material. The specific rotation is given by $\rho = (CB)/d$. At $\lambda = 560$ nm, we find $|CB| = 0.37$ radians (21°) and with a thickness of 20 $\mu$m we have 525°/mm compared to 560°/mm earlier found at $\lambda = 562$ nm in $C. aurata$.

In the related work, we found a cuticle thickness of 20 $\mu$m that is used here as an estimate. The actual value is of minor importance as it cancels out. This is due to that it enters as a fixed parameter in the regression. An error in thickness will then cause an error in the model parameter $\kappa$. When $\kappa$ is calculated from $CB$ and $CD$, the thickness enters in Eq. (5) and the effect of an error is eliminated to first order. The effect will be that there is an
uncertainty in the absolute values (scale uncertainty) in Fig. 5. The error is estimated to be <10%, but it is of minor importance as the objective with this communication is to compare determination of chirality using a model-free approach with the use of an electromagnetic model-based approach.

The cuticle materials are dielectric and nonabsorbing, but a cuticle is locally inhomogeneous and some scattering may occur leading to apparent absorption. However, due to the use of a normalized Mueller matrix, both electromagnetic modeling and differential decomposition will be unaffected by this. No isotropic absorption is, therefore, included in the electromagnetic modeling. However, the differential decomposition reveals some dichroism that is added with an Urbach tail dispersion to the electromagnetic model to improve the fit. This is not Kramer–Kronig consistent with the Cauchy dispersion for the refractive index. However, the physical origins of $LB$ and $LD$ are most probably different.

The structure is generally believed to be a twisted layered structure composed of biaxial layers arranged in multiple turns. With a pitch of around 400 nm\textsuperscript{13} and a cuticle thickness of 20 $\mu$m, there are around 50 turns. If the number of half turns is an integer, the linear birefringence and dichroic effects would cancel out in an effective medium approach that is used here. The small but nonzero values of $LB'$ and $LD'$ are most likely due to that the number of half turns deviates from an integer resulting in net linear anisotropy.

The $CD$ spectrum in Fig. 4 shows a shoulder on the long-wavelength side of the Bragg resonance. This indicates that there are two helicoids with different pitches in the cuticle, which also is confirmed by the need of two Gaussian resonances in the regression analysis of the data. Multiple pitches are plausible as the beetle cuticle can exhibit complex pitch variations across the cuticle as found by optical mode analysis and electromagnetic modeling.\textsuperscript{23–25} From the present data, we cannot determine whether the two resonances stem from two lateral structures or two structures at different depths, i.e., if they are in parallel or in series. In electromagnetic modeling using reflection Mueller matrices, we found that a smearing of the pitch was needed,\textsuperscript{13} but in the present case, two distinct resonances are more probable as smearing would broaden the resonance rather than result in a shoulder.

Small resonancelike features are observed in the primary data in Fig. 2 in many of the off-diagonal elements. These features propagate through the differential decomposition and can be seen in $LB$, $LD$, $LB'$, and $LD'$ in Fig. 4. They occur in the same spectral region as the Bragg resonance. The chiral model with an addition of anisotropic dispersion and absorption can reasonably reproduce baseline effects in the linear properties but not the features related to the Bragg resonance and the small ripples observed in the experimental data in Fig. 3 as well as in the results in Fig. 4. Those features are due to that the cuticle is a multilayer consisting of lamellae, all composed of the same anisotropic material and rotated from lamella to lamella. Consequently, the light experiences small differences in the refractive index when travelling between lamellae, which creates interference effects within the cuticle resulting in the resonance feature and the spectroscopic ripples in the linear properties. The model based on a single layer cannot reproduce these features because of the absence of a stack of layers. Nonetheless, the experimental data can be successfully fitted with the simple chiral layer because the linear effects are much smaller than the circular effects. To further support this explanation of the origin of the features in the linear properties, a differential decomposition was performed on simulated Mueller matrix transmission data generated from a structural model developed for $C. aurata$.\textsuperscript{13}

As seen in Fig. 6, the features in the linear properties appear at the Bragg resonance wavelength. The ripples are very weak in this simulation but can be seen on an expanded scale.

FIG. 6. Differential decomposition of transmission Mueller matrix data obtained by forward calculations using a structural model based on twisted anisotropic multilayers used in electromagnetic modeling of reflection Mueller matrix data (Ref. 13).

V. SUMMARY AND CONCLUSIONS

Nonlinear regression and differential decomposition are performed on the same transmission Mueller matrix from a beetle cuticle with objective to determine chirality of a circular Bragg structure. The cuticle medium was assumed to be a homogeneous bianisotropic medium. The two methods gave very similar results. Differential decomposition is model-free and all information is preserved, but the method requires in-depth homogeneity. Nonlinear regression requires dispersion models that may lead to incomplete modeling. However, it can be applied to inhomogeneous systems, e.g., graded layers and multilayered structures.

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