

Understanding lidar returns from complex dust mixtures

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ABSTRACT

Lidar is a powerful tool for measuring the vertical profiles of aerosols. Dusts are irregularly-shaped particles with varied composition and strong variations in index of refraction in the LWIR. We measure dust indices using ellipsometry and transmission through KBr pellets. Milling makes the ellipsometry data less dependent on incidence angle, and the results of measurements on milled materials agree with those from transmission measurements. Measurements show that the spectrum of a milled Arizona Road Dust (ARD) approaches that of pure quartz, indicating that the decrease of absorption efficiency for particles larger than the absorption length substantially affects the results. These indices of refraction are intended for future simulations of extinction for LWIR lidar beams.

Keywords: aerosol scattering, ellipsometry, optical transmission, effective medium theory, index of refraction measurement

1. INTRODUCTION

Measurements of the index of refraction of aerosol are challenging and prior measurements show large disparity in the results [1-4]. Lidar data analysis necessitates an improved understanding of the optical properties of airborne particulate matter [5-10]. We describe a new approach for measuring these properties that significantly improves our ability to measure the complex index of refraction. Scattering calculations, whether Mie, T-matrix calculations, or a direct solution of Maxwell's equations, require the index of refraction as an input parameter [11-15]. Neither the optical extinction nor the scattering phase functions can be obtained without the indices. The obvious method to measure the index of refraction involves collection of the aerosol particulates to form a pellet for reflectivity measurements of the complex optical refraction indices. This method is confounded by scattering of the light and by the physical structure of the particulate. Milling the particles until they are small, and more uniform, relative to the wavelength can minimize the effects of particle scattering in the sample. The magnitude of the scattering from individual particles in a pellet sample is minimized by choosing a host material with a real index of refraction similar to that of the particulate to be studied. However, it does not completely eliminate scattering in the regions of very large gradients in the indices of refraction, such as near a sharp absorption resonance, or for measurements at wavelengths near or below the size of the smallest particulates after milling. When we claim to measure the index of refraction, we mean the bulk index. This is the quantity used in scattering calculations, but the (bulk) index of refraction is only a valid concept deep inside a large volume of material. Near the surface, the particular atom or molecule reacts to the bound surface charge at the interface between the materials, as required by Maxwell's equations. Thus, the effective local index of refraction is changed. We show that it can shift the wavelength at which sharp absorption features are observed, thereby precluding the use of a simple linear combination of material dielectric constants in determining the particle refractive index. Thus, the effects of the proximity to the surface of nearly all atoms in a small particulate must be corrected as part of the data analysis.

The technique used for the refractive index correction is based upon effective medium theory [16]. It is valid when the sizes of the particles are less than about 1/10 the wavelength of the light. Calculations require that the volume fraction of the components of a mixture be determined to obtain the correct optical properties (including air, if it is the host medium for the particles). Several possible variants of effective medium theory have been devised for different scenarios. Maxwell-Garnett theory is useful for single constituent particles embedded in a medium but well separated from each other. The Bruggeman effective medium approximation is useful for coated particles or material mixtures in which there is no clear choice of host material over inclusions [17, 18]. The limits of the possible dielectric constants can be evaluated with minimum and maximum shielding effective medium models. We show here that use of effective medium theory is required for accurate determination of the index of refraction. We also show that Mie scattering calculations include effective medium corrections, as they must, since they are based directly on solutions to Maxwell's equations [e.g. 10].

In this paper, we evaluate the usefulness of transmission measurements for obtaining the complex index of refraction of particles. By taking the ratio of the transmission of two pellets with the same composition but different thicknesses, we can eliminate most of the uncertainties of sample reflectivity, the absorption of surface films, and greatly simplify the data analysis. The imaginary index is easily obtained from the ratio of data obtained with different thickness pellets, and the real index follows from a Kramers-Kronig analysis [19]. Effective medium theory is then used to determine the complex index of refraction of the particles. We compare the transmission results to those indices of refraction determined by ellipsometry, and find good agreement at long wavelengths and away from sharp resonances. We also find that the particles must be milled prior to making ellipsometry measurements; otherwise, the indices determined by ellipsometry are dependent upon the angle of incidence of the light, which is obviously not physically correct.

2. ELLIPSOMETRY OF PRESSED ARD

2.1 Sample preparation

Ultrafine ARD particles (PTI – Power Technology Inc.) were placed in dry air at a temperature of 70 °C overnight to remove any absorbed water. A sample of 300 mg of dust was then placed into an International Crystal Laboratories pellet die. The pellet die was then evacuated to approximately 10 mTorr vacuum and a load of 8 tons of force was applied for 5 minutes. The anvils that compressed the dust were optically polished stainless steel. This produced ½-inch diameter pellets of approximately 1.5 mm thickness with nominally optically flat surfaces suitable for ellipsometry. Another sample of dust was first milled for 4 hours in a SPEX 1500 Mixer/Mill with Tungsten Carbide grinding balls and end caps to make a more uniform sample with an average particle size below 1 micron; this process provides better packing and a smoother surface. After milling, the samples were prepared as described above. After pressing, the pellets are extracted from the pellet press and immediately adhered to a glass microscope slide with double stick tape. The pressed pellets are robust in the normal direction but are highly susceptible to shear stress. The edges of the pellets are painted with silver paint to make them stronger, and allowed to dry. This produced robust pellets that could be vertically mounted in our ellipsometer.

2.2 Ellipsometric Measurement

A J.A. Woolam Co. IR-VASE ellipsometer, with a nominal spectral range of 1.7-30 microns, is used to measure the optical constants of our dust pellets. The ellipsometer is set to a resolution of four wavenumbers per step and a total of 200 scans are averaged for each angle of incidence. Each incidence angle measurement takes approximately 10 hours. Figures 1 (a) and (b) show experimentally determined n and k from ellipsometric measurements of as received ARD at 3 angles of incidence (60, 65, and 70 degrees). The complex index $n + ik$ are derived using a direct inversion of the single reflection model given by,

$$\epsilon_r + i\epsilon_i = \sin^2(\phi) \left[1 + \tan^2(\phi) \left(\frac{1-\rho}{1+\rho} \right)^2 \right],$$

where ϵ_r is the real component of the dielectric function, ϵ_i is the imaginary component, ϕ is the angle of incidence, and ρ is a function of the ellipsometric parameters Ψ and Δ given by,

$$\rho = \tan(\Psi) e^{i\Delta}.$$

The equations for the relationships between the complex dielectric function and n and k are,

$$n = \sqrt{\frac{\sqrt{\epsilon_r^2 + \epsilon_i^2} + \epsilon_r}{2}}$$

$$k = \sqrt{\frac{\sqrt{\epsilon_r^2 + \epsilon_i^2} - \epsilon_r}{2}}.$$

Figures 1(a) and b) show the n and k indices of as-received Ultrafine ARD plotted as a function of varying incidence angle, although they should be independent of the incidence angle. This is likely due to either surface roughness effects or reflections from the back surface of the pellet. Also, IR transmission measurements of aerosolized ARD do not show the absorption peak at 7.6 microns [4]. Therefore, it is assumed that the peak is not a part of the dust itself but is likely due to the double stick tape which is in intimate contact with the backside of the pellet. Figures 2 (a) and (b) show the real and imaginary index of a pellet formed from the milled ARD. These measurements have no incidence angle dependence on n and k .

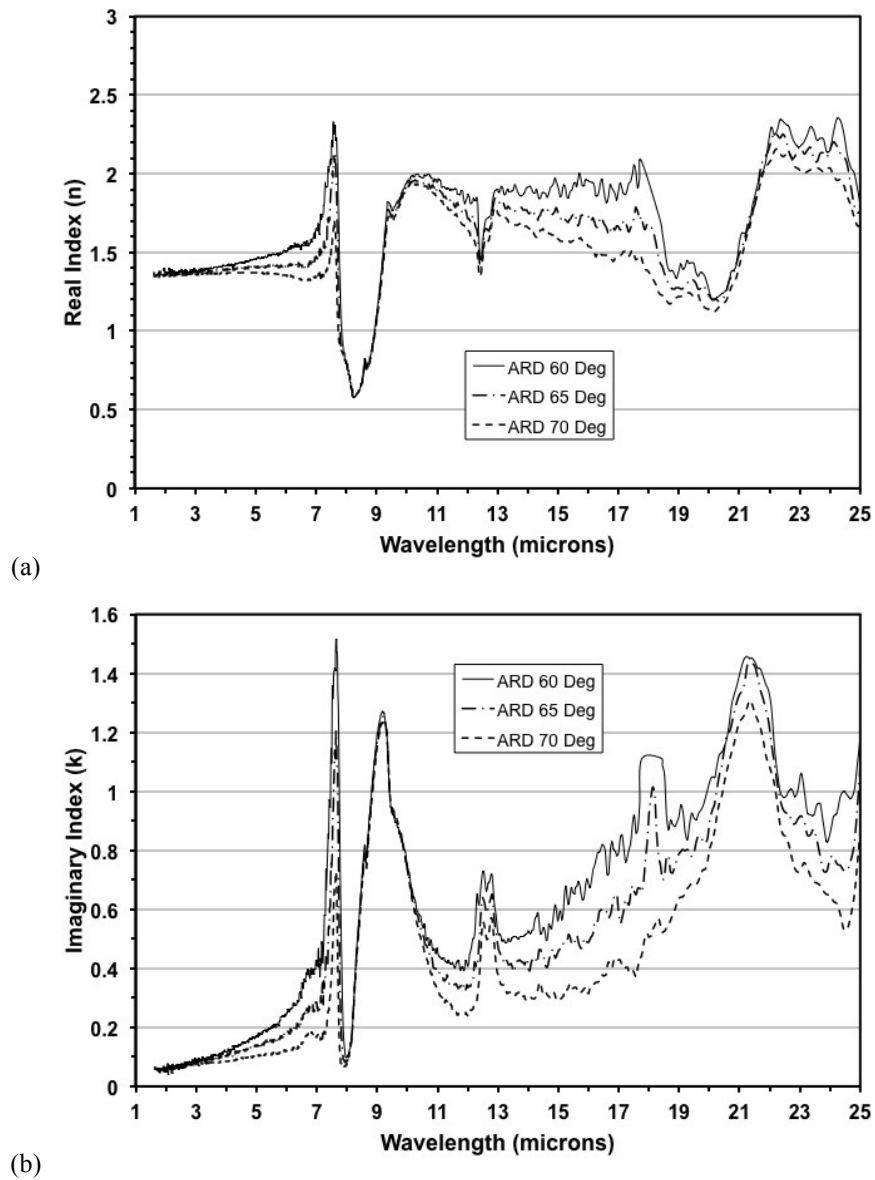
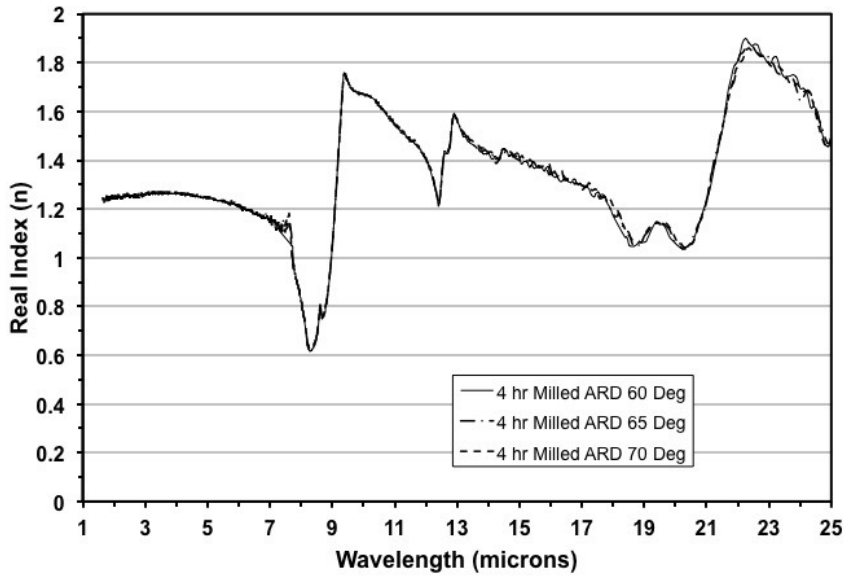
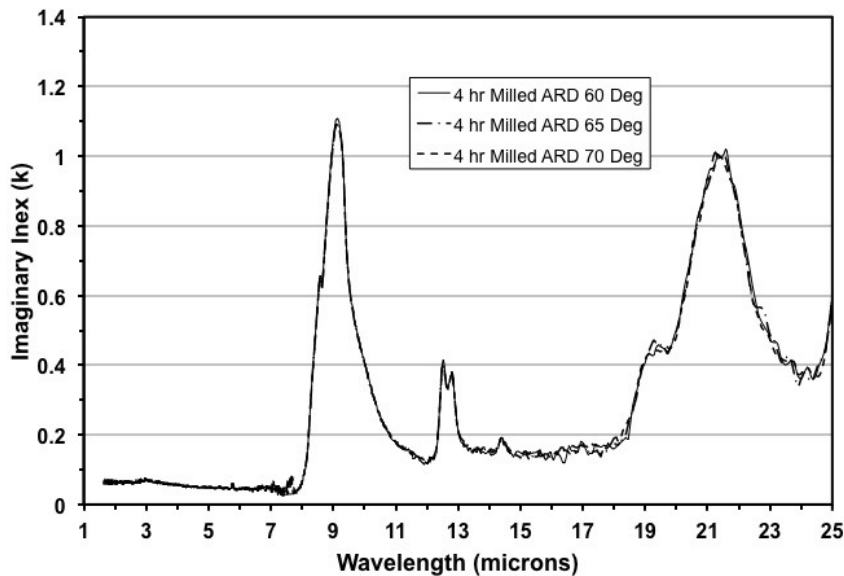


Figure 1. Ellipsometry determined n and k for as-received Ultrafine ARD; (a) Ultrafine ARD real index of refraction is different for different angles of incidence; (b) imaginary index of Ultrafine ARD shows an erroneous peak at 7.6 microns as well as incidence angle dependence.



(a)



(b)

Figure 2. Ellipsometry determined n and k for Ultrafine ARD that is milled prior to being pressed; (a) the real index of a milled ARD pellet the data shows low noise and excellent agreement at various incidence angles; (b) the imaginary index once again it shows low noise and no apparent angular dependence; moreover, the erroneous 7.6 micron peak does not appear.

It was, therefore, determined that to make reliable ellipsometry measurements of dust aerosols, the particles must first be ground to a small size and pressed into pellets to make smooth, specularly reflective surfaces. The pellets must pack tightly enough to prevent air pockets. This process also appears to eliminate the artificial 7.6 micron absorption.

3. TRANSMISSION MEASUREMENTS OF ARD

In the previous section, we found that ellipsometry measurements are sensitive to surface effects and particles sizes when samples are prepared with powders pressed into pellets. A transmission measurement is less sensitive to surface effects, as the entire bulk of the sample is traversed. Therefore, we investigate the measurements of the optical properties of ARD with transmission of pellets when the particles are suspended in a matrix of KBr. This technique has been used extensively in the past for molecular measurements; however, it does not appear that any of the previously reported measurements adequately accounted for effects from interaction with the surrounding media.

3.1 Sample Preparation

The basic requirement to determine the optical properties of particles from transmission measurements is that particle sizes must be smaller than the wavelength. In fact, a stricter bound, $r < \lambda/10$, was proposed by Aspnes [16,17]. This ensures that the extinction of light passing through the suspended aerosol is primarily due to absorption, and not an unknown combination of absorption and scattering (any remaining Rayleigh scattering is small and assumed negligible). The ARD size distribution, provided by PTI shows a significant number of particles with a radius larger than 5 μm . Therefore we mill the dust to enable successful ellipsometry measurements. This ground dust is stored in dry air overnight along with powdered KBr (International Crystal Labs). The KBr and milled ARD are then weighed to give a mass percent of 0.48% ARD to KBr. PTI reports the density of ARD to be 2.65 g/cm^3 and the density of KBr is 2.75 g/cm^3 , therefore, this mass percent corresponds to a volume percent concentration of 0.5% ARD in KBr. The two samples are mixed in an agate mortar and pestle for 2 minutes and then placed back into the oven to keep dry until pressed into pellets. Various amounts of the KBr-ARD mixture are weighed (100 mg-300 mg) and placed into the $\frac{1}{2}$ inch diameter pellet die, which compresses the mixture evenly under 10 mTorr vacuum, with 8 tons of force for 30 minutes. Different weights of the mixture create pellets of different thickness to simplify the analysis, which will be described below. After pressing, the pellets are found to be clear and have optically flat surfaces when they are extracted from the die set.

3.2 Converting Transmission Measurements to n and k

The conversion of transmission measurements to indices of refraction has many possible pitfalls. First, reflections from the two surfaces of the pellet mixture must be considered. The total transmission of an absorbing pellet in air is given by the equation,

$$T = (1 - R_1)(1 - R_2)e^{-\alpha d} \quad (1)$$

where R_1 is the reflection of light in air incident on the front surface of the pellet, R_2 is the reflection from the back surface, α is the extinction coefficient of the mixture, and d is the thickness of the pellet. The reflections from each of the surfaces are given by Fresnel's equations for light encountering an interface between air and the medium with complex index of refraction $n + ik$ at normal incidence,

$$R_1 = \frac{(1 - n)^2 + k^2}{(1 + n)^2 + k^2}, \quad R_2 = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \quad (2)$$

The third term of Eq. 1 is the Beer-Lambert law for absorption of light through a medium. It is dependent on only the extinction coefficient (itself a function of the wavelength and the imaginary index of the medium, assuming no scattering) and the thickness through which the light propagates. The relationship between the extinction coefficient, α , and the imaginary index of refraction is given by the equation,

$$k = \frac{\alpha \lambda}{4\pi}$$

It is evident that neither n nor k can be directly extracted from a single transmission measurement. In previous measurements, it was assumed that the real indices of the KBr and the dust were close and that the imaginary index of the dust inclusions did not significantly affect the reflection at the surface [20-22]. These assumptions are reasonably good when the volume fraction of the dust is small; however, they are still significant near absorption resonances of the dust in the KBr. We solved this problem by making transmission measurements on two pellets of different thicknesses, then taking the ratio to eliminate the reflection effects. The surface reflections should be identical since the dust concentrations are the same, so the only remaining quantity from the ratio (Eq. 1) is the ratio of absorption components,

$$\frac{T_1}{T_2} = \frac{e^{-\alpha d_1}}{e^{-\alpha d_2}} = e^{-\alpha(d_1-d_2)}$$

In this equation T_1 is the transmission of pellet 1, T_2 is the transmission of pellet 2, d_1 is the thickness of pellet 1, d_2 is the thickness of pellet 2, and α is the extinction coefficient, as before. The equation for extraction of the imaginary index of the mixture from the transmission ratio is given by,

$$k = \frac{\lambda}{4\pi(d_1 - d_2)} \ln\left(\frac{T_1}{T_2}\right),$$

where T_1 , T_2 , d_1 , and d_2 are defined above and λ is the wavelength of light. Values of n can then be determined by a Kramers-Kronig analysis, as described by Wagner [19], where the short wavelength n in the integral is determined by an effective mixture of the major component of dust aerosols (i.e. silica) and KBr.

3.3 Transmission Measurements

Transmission measurements are made on the same IR-VASE (J. A. Woolam Co.) that is used to make the ellipsometry measurements, but setup in transmission mode. The resolution is set to 4 wavenumbers with 50 scans averaged per measurement. Figure 3 shows n and k of 0.5% ARD in KBr pellets derived from transmission. The left axis is the real index and the right axis is the imaginary index. The low concentration of road dust in the KBr only causes a small variation in the real index of the KBr and results in a very low absorption index. Therefore, the impact of surface reflections due to the dust is not a large effect but their removal from the determination of k is the correct approach. To extract the refractive index of the dust inclusions, the usual approach is simply to take a volumetric average of the constituents [20-22]. However, this only works if the indices of the inclusions are slowly varying as a function of wavelength. If the indices change markedly like those of silica, and silica containing substances, then the effective medium approach must be considered. Otherwise the peaks in the imaginary index can be shifted along the wavelength axis by 10%, as illustrated in the following description.

3.4 Effective Medium Analysis of Transmission Data

When particle inclusions are mixed into a matrix material, the dielectric properties, and therefore the real and imaginary indices of the matrix material, are perturbed by screening charges that accumulate at the interfaces between the inclusion and the surrounding material [16]. Effective medium theories are used to describe these effects averaged over the entire material. For isolated or sparsely dispersed (<5%) spheres in a host material, the resulting effective complex dielectric constant can be found using the Maxwell-Garnett Equation,

$$\frac{\epsilon - \epsilon_h}{\epsilon + 2\epsilon_h} = f \frac{\epsilon_b - \epsilon_h}{\epsilon_b + 2\epsilon_h},$$

where ε is the effective complex dielectric constant, ε_h is the complex dielectric constant of the host matrix, ε_b is the complex dielectric constant of the spherical inclusions, and f is the volume fraction of inclusions in the host matrix.

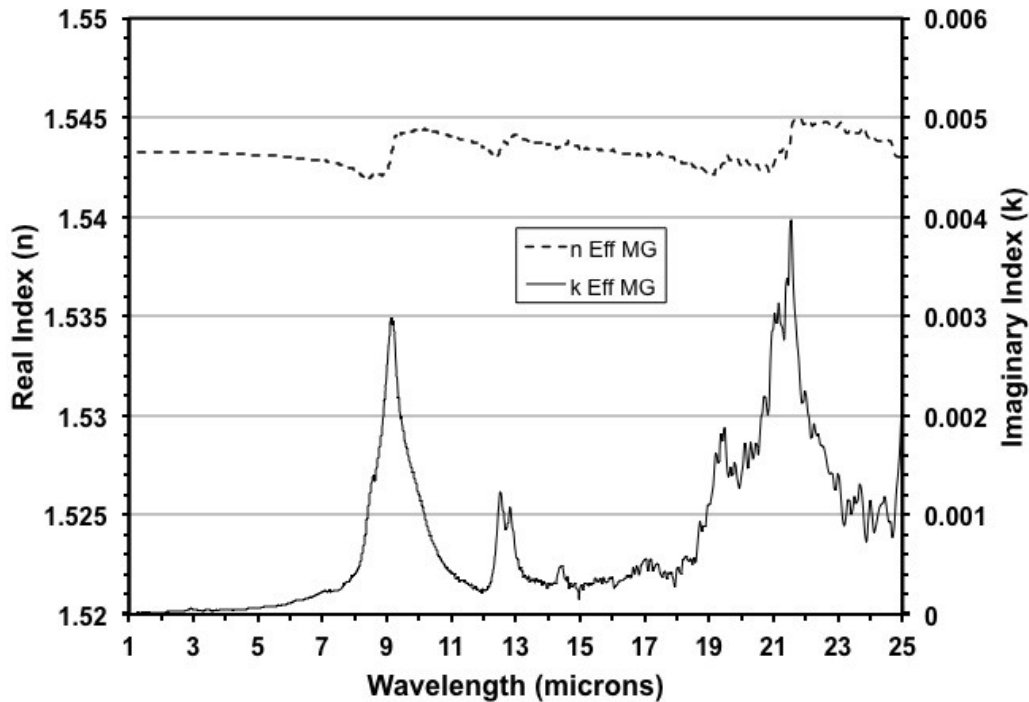


Figure 3. Indices of refraction of 0.5% ARD in KBr pellets from transmission the low concentration of ARD in KBr causes only a slight variation in the real index from that of KBr itself, and a very small absorption index.

If the dielectric constant of the host material is known, the inclusion volume fraction is known, and the effective dielectric constant (complex refractive index) is measured through transmission, it is possible to solve the Maxwell-Garnett Equation to extract the complex refractive index of the inclusions. As a demonstration of the effect on the measured indices of refraction by an interaction between a matrix and its inclusions an effective medium calculation of 0.005 v/v silica suspended in air is shown in Figures 4 (a) and (b), which compare the effective real and imaginary indices of the mixture, with those of silica. As can be seen from the figures, the peaks and transitions of the effective indices are shifted to the left from those of pure silica --- this has important implications. The common method of using a volume fraction weighted linear combinations of the component indices cannot be used to retrieve silica's indices from the observed effective indices. A linear volume mixing model for a surrounding medium, such as air or KBr, that have a constant or slowly varying real index and no significant imaginary index over the observed wavelengths, cannot produce a left or right shift in features in the effective refractive index. Air was chosen in this demonstration to amplify the peak shift. If KBr had been chosen, the peak shift would still exist; however, it would be only ~4% because of the better index match between KBr and silica.

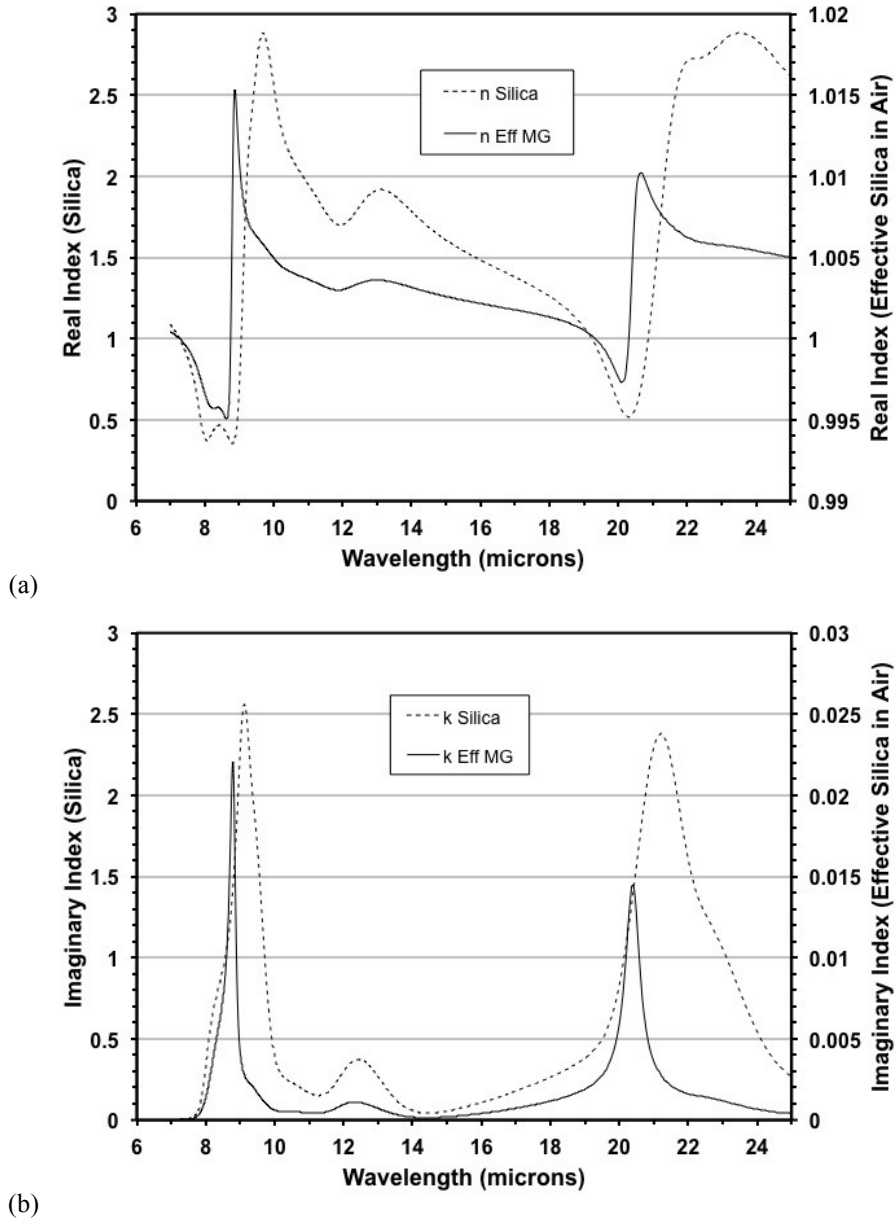
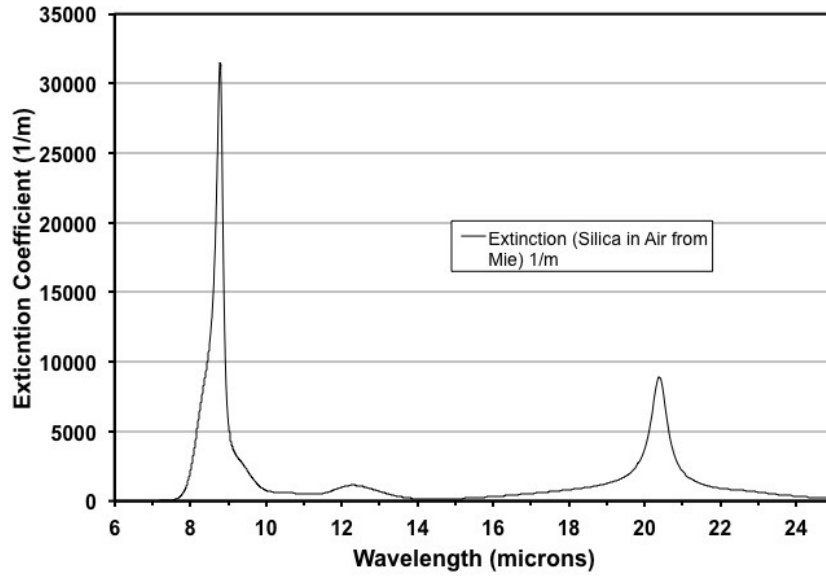
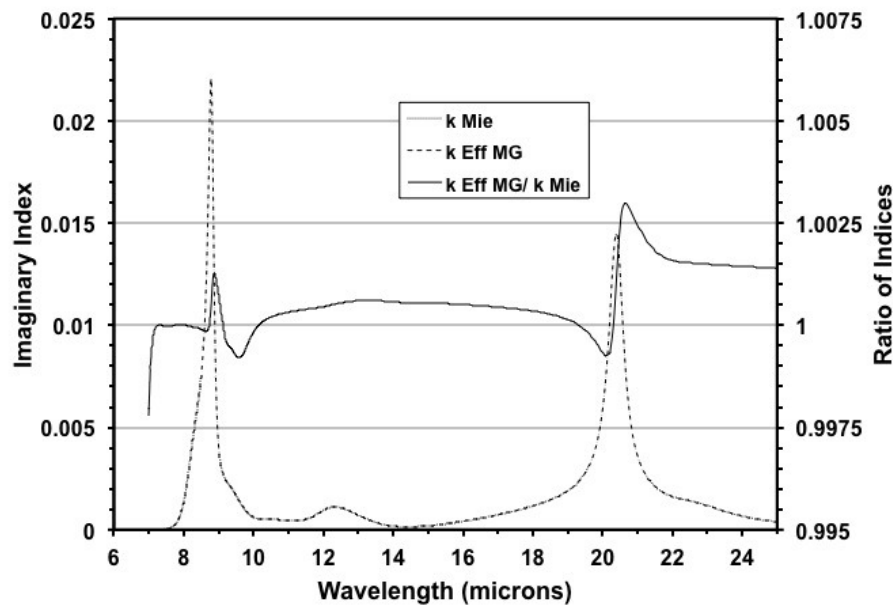


Figure 4. Comparison of n and k of 0.005 v/v silica (right axis, narrow range) in air versus pure silica (left axis); (a) the transitions in n associated with the peaks in the imaginary index are shifted to the short wavelength side by approximately 10%; (b) the effective imaginary index of silica in air also shows a significant peak shift, and the absorption bands of this composite material would therefore be shifted to shorter wavelengths than would be observed for pure silica.

We modeled 0.005 v/v 100 nm silica spheres in air using Mie calculations for wavelengths from 6-25 microns to further demonstrate that transmission measurements of absorption peaks can be shifted by an interaction with the surrounding medium. Figure 5(a) shows the extinction coefficient, α , as a function of wavelength derived from the Mie calculations. Figure 5(b) shows the index of refraction derived from the Beer-Lambert law relationship between α and k compared to the effective imaginary index of 0.005 v/v silica in air. The Mie calculation derived imaginary index and the effective medium derived imaginary index are virtually identical. The ratio of the k derived from Mie calculations and that derived from effective medium are plotted along with the indices on the right axis. The maximum variation is approximately 0.25%.



(a)

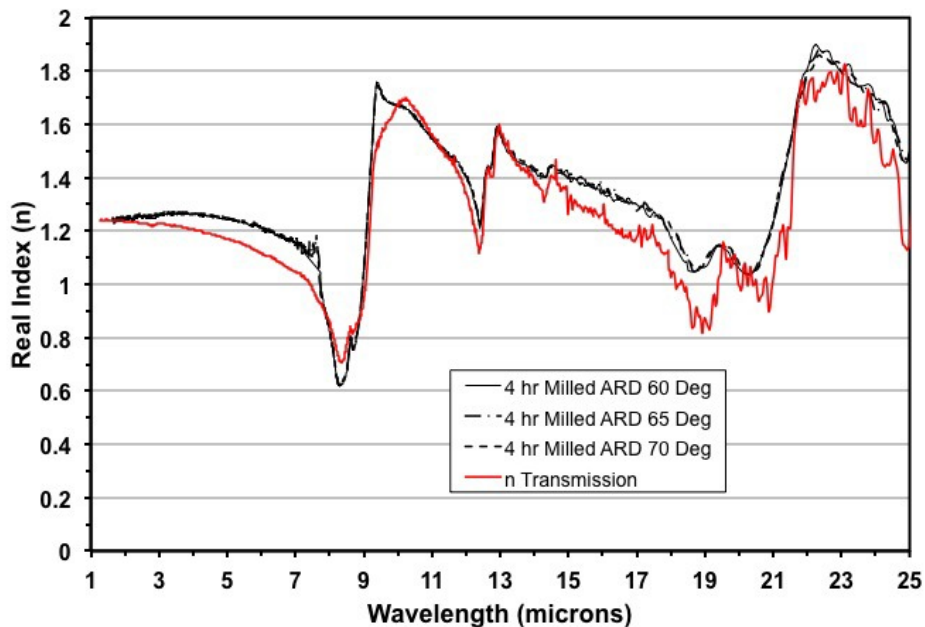


(b)

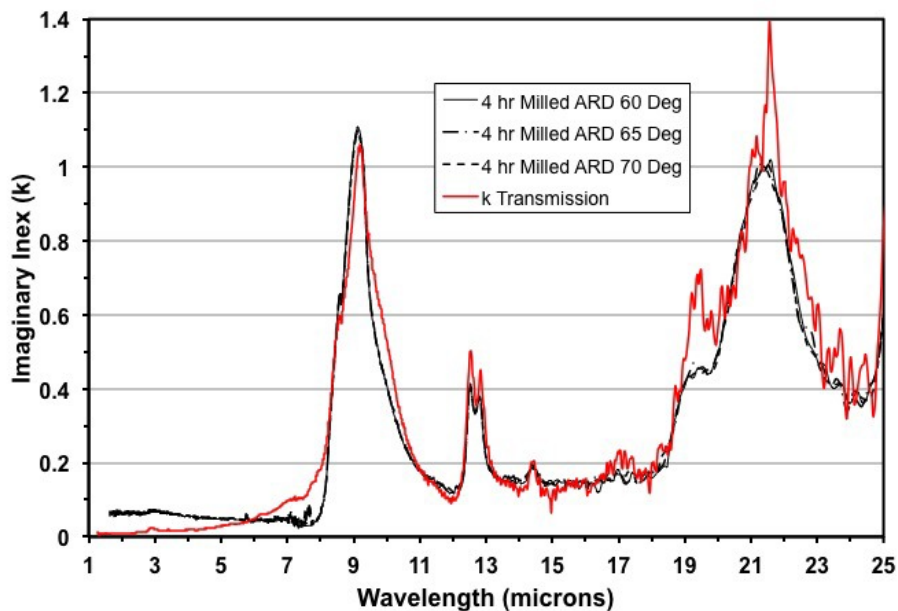
Figure 5. Mie calculation of extinction is compared with result from effective medium; (a) wavelength dependent extinction coefficient of 0.5% by volume silica in air from Mie calculations; (b) comparison of the effective imaginary index of silica in air with that derived from Mie extinction agree to within the line width (left axis). The ratio of the two (right axis) indicates that the variations are at most 0.25%

The effective medium approximation is used to extract the indices of the milled Arizona road dust from the measured indices of the ARD and KBr mixture pellet. The results are compared with those measured with ellipsometry in Figures 6 (a) and (b), showing the real and imaginary indices of the ARD compared with ellipsometry measurements, and also for milled ARD. Very good agreement is seen between the imaginary indices above 8 microns. The disagreement below 8 microns is likely due to particle size effects and scattering. There is a larger discrepancy, on the order of 10%, between the real indices measured with ellipsometry and those derived from transmission. These errors can arise from scattering,

imperfections in the KBr pellet, and errors in the assumption of n at small wavelengths (part of the Kramers-Kronig process).



(a)



(b)

Figure 6. Comparison of transmission derived indices of milled ARD with those derived from ellipsometry; (a) the real index as a function of wavelength; (b) the imaginary index as a function of wavelength. Very good agreement is seen between the imaginary indices above 8 microns however a 10% difference is seen for the real component.

CONCLUSIONS

An experimental study of two methods of determining the optical properties of Arizona Road Dust has revealed that, when treated correctly, transmission measurements of sparse dust in a matrix of KBr and ellipsometry measurements of pure dust pressed in pellets do agree quantitatively. Transmission measurements are faster to conduct, require far less dust to make samples than ellipsometry, and use cheaper equipment; however, transmission measurements require small particles and a Kramers-Kronig analysis to retrieve an effective n . It also requires an effective medium analysis to isolate the dust inclusion indices from those of the KBr/dust mixture to find the effective index. Ellipsometry measures both the real and imaginary index at the same time, but it requires milling of dust to produce uniform pellets with optically flat surfaces so that the results are independent of incidence angle. It is highly susceptible to surface imperfections and films, requires exceptionally expensive equipment, and requires a long time to make a low noise measurement of dust in the infrared. Our analysis approach demonstrates that accurate measurements of the refractive indices of single component particulate distributions can be made via transmission, for ranges of wavelengths several times larger than the diameter of the milled particulates.

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