Facile Method for Determining the Aspect Ratios of Mineral Dust Aerosol by Electron Microscopy

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Mineral dust is the second largest atmospheric emission by mass and one of the least understood sources. The shape of the particles depends on their composition and has implications for particle optical properties and reactive surface area. Mineral dust particles are often approximated as spheroids to model their optical properties. In this study, scanning electron microscopy (SEM) is used to measure the aspect ratios of calcite, quartz, NX-filite, kaolinite (KGa-1b and KGa-2), and montmorillonite (STx-1b and SVWy-2). In addition to traditional SEM images of the top of the particles, the SEM substrates are oriented approximately normal to the electron beam in order to image the side of the particles. In this manner, aspect ratios for the top and side orientation of the particles are determined. Calcite particles have an aspect ratio of approximately 1.3 in both orientations, while quartz particles have an aspect ratio of 1.38 in the top orientation and 1.64 in the side orientation. The clay minerals studied all exhibited plate-like structures with aspect ratios of 1.35 to 1.44 for the top orientation and 4.80 to 9.14 for the side orientation. These values are used to estimate the specific surface areas (SSAs) of the minerals, which are compared to Brunauer-Emmett-Teller (BET) surface area measurements. Through this study, we present a simple method for determining the aspect ratios of aerosolized samples, rather than relying on literature values of model systems. As a result, this technique should provide a better method for determining the optical properties of mineral dust particles.

INTRODUCTION

Aerosol particles affect the radiative balance of the Earth through their interactions with light and clouds as well as their influence on atmospheric composition (Forster et al. 2007). How particles scatter and absorb light is determined by their shape, composition, and size. Mineral dust aerosol particles also act as nuclei for liquid water and ice clouds (Broadley et al. 2012; Pinti et al. 2012; Atkinson et al. 2013). In addition, heterogeneous chemistry on mineral dust aerosol particles can alter atmospheric composition (McNaughton et al. 2009; Fischer et al. 2010).

Mineral dust aerosol particles are the second largest emission by mass into the atmosphere, amounting to 1000 to 3000 Tg per year (Dentener et al. 1996; Ginoux et al. 2001; Forster et al. 2007; Ginoux et al. 2012). A major source of mineral dust comes from dust storms in arid regions that entrain large amounts of particulates in the atmosphere, which can subsequently be transported long distances from the source region (Cahill 2003; Zhao et al. 2003; Fairlie et al. 2007). Up to 50% of mineral dust emissions originate from anthropogenic sources such as agriculture (Forster et al. 2007; Ginoux et al. 2012).

Aluminosilicate clay minerals are one of the most common types of mineral dust, accounting for 50–64 wt.% of Saharan dust (McNaughton et al. 2009) and up to 85% of Asian dust (Liu 1985). Mineral dust is one of the most common ice nuclei, and aluminosilicate clay minerals are one of the most ice-active types of mineral dust (Broadley et al. 2012; Pinti et al. 2012). Ice nuclei from field samples have been studied in great detail using scanning electron microscopy (SEM) and scanning transmission X-ray microscopy (STXM; Wang et al. 2012; Hiranuma et al. 2013). Among mineral dust components, clay minerals have the largest aspect ratios, with disk-like shapes and measured aspect ratios of approximately 4 to 500 (Nadeu 1985; Inoue and Kitagawa 1994). These shapes cause aluminosilicate clay minerals to have different optical properties than particles that have aspect ratios closer to unity. In addition, these particles have a large surface area to volume ratio because of their large aspect ratio. Because they have a larger surface area to volume ratio than other types of particles, they are more active toward ice nucleation and heterogeneous chemistry per unit volume.

Clay minerals are composed of negatively charged aluminosilicate layers composed of alternating layers of tetrahedrally coordinated silicon and octahedrally coordinated aluminum, resulting in alternating tetrahedral and octahedral layers. Al$^{3+}$ can be substituted into the tetrahedral layers and divalent cations (e.g., Mg$^{2+}$) can be substituted into the octahedral layers, which results in the net negative charge.
Monovalent cations (e.g., Na\(^+\) and K\(^+\)) and calcium tend to sit in the interlayer sites, which holds the aluminosilicate layers together through electrostatic interactions (Moore and Reynolds 1997). The number of tetrahedral to octahedral layers, for example, 1:1 or 2:1, defines the particle type (Martin et al. 1991). For example, kaolinite is a 1:1 clay, while montmorillonite and illite are 2:1 clays that vary in the number and composition of interlayer ions (Grim 1968).

Two other major components of mineral dust are quartz and calcite. Quartz is the largest component of China Loess, at greater than 50 wt.% (Liu 1985). Calcium carbonate is a highly reactive component of mineral dust and a major fraction of Asian dust, contributing up to 12 wt.% (Liu 1985; McNaughton et al. 2009). A study of Asian dust collected over the Eastern North Pacific estimated that 5–30% of calcium carbonate was converted to CaSO\(_4\) or Ca(NO\(_3\))\(_2\) during transport (McNaughton et al. 2009).

Current uncertainties in the radiative impacts of mineral dust aerosol include size distributions, particle shape, and composition-resolved emission concentrations (Johnson et al. 2009, 2012; Kok 2011). Both satellite and ground-based lidar measurements use algorithms that include particle shape to accurately derive aerosol mass loadings. Traditionally, shapes of particles in models are estimated by spheroids, where the axes of the spheroid are determined by the particle aspect ratios (ratio of the width to the length or height of the particle). By using a distribution of particle aspect ratios, the radiative properties of particles have been more effectively modeled in aerosol retrieval algorithms (Haapanala et al. 2012). Many studies have found that incorporating the shape distribution with a large range of aspect ratios has improved models, but lack a physical reason for their choice of aspect ratios (Kahnert 2004; Nousiainen et al. 2006; Merikallio et al. 2011). Hudson et al. (2008) found that incorporating the shape of clay mineral particles (e.g., disc/needle) improves the agreement between models and laboratory results. Recently, Lindqvist et al. (2014) used SEM to characterize the shape and composition of supermicron mineral dust aerosol particles. They found a large difference between the scattering properties of spheres, spheroids, and particles with the measured shapes and compositions (Lindqvist et al. 2014).

For the minerals of interest in this study, two aspect ratios are defined in reference to the substrate on which they are placed for this measurement. One aspect ratio is the ratio of the length to the width of the top of the particle (top-down orientation), and one is measured as the ratio of the length to the height of the side of the particle (side-on orientation). The axes are chosen such that the aspect ratio is greater than unity. For the larger of the two aspect ratios, it has been shown that calcite has an aspect ratio near 1, quartz has a slightly higher aspect ratio near 1.7–1.9 (Siegesmund et al. 2002), and clay minerals have aspect ratios from 3 to approximately 500 (Nadeau 1985; Inoue et al. 1994). Previous studies have determined the high aspect ratios of clay minerals using transmission electron microscopy (TEM) and atomic force microscopy (AFM). To measure the aspect ratio using TEM, particles are coated (generally with Pt) at a known angle, approximately 10 degrees from normal. Due to the angle of the coating process, the height of the particle shields part of the substrate from being coated. The height of the particles can be calculated based on the width of the uncoated region. Using the two dimensional projection of the particles visible in the TEM image and the height of the particle, the aspect ratios of the particle in the top-down and side-on orientations can be determined (Robertson et al. 1954; Jepson and Rowe 1975; Nadeau 1985, 1987; Inoue and Kitagawa 1994; Becket et al. 1997; Tumolva et al. 2012). This method assumes that the particle has no surface irregularities that may affect the observed height of the particle and that the particles do not move during the coating process. This technique has been used extensively to determine the volume of mineral dust particles. Aspect ratios for clay minerals have been as high as 1.68 for montmorillonite, 1.31 for kaolinite, and 5.33 for illite for the top-down orientation and as high as 448, 18, and 120, respectively, for the side-on orientation (Nadeau 1985). AFM has also been used to measure the dimensions of clay minerals without the use of a coating (Lindgreen et al. 1991; Schleicher et al. 1993; Bickmore et al. 2002; Tumolva et al. 2012). In addition to microscopy methods used to study the dimensions of the particles, surface area measurements through gas adsorption studies (described below) can be used with microscopy methods to find the dimensions of the particles (Hofmann et al. 1961; Shuttlefield et al. 2007). Because of the need for a coating for the TEM measurement of the side aspect ratio and the nature of the AFM technique, these methods are slow. As a result, they are not used routinely to study field and laboratory samples. Instead, in studies where aspect ratios are needed for analysis, literature values are used, which may not be representative of the exact sample of interest. The development of techniques to easily measure aspect ratios for samples would help our analysis of field and laboratory samples because each sample of interest could be measured rather than depending on literature values for representative compounds. Where additional information is needed for complex particles, more time-consuming techniques such as electron tomography and focused ion beam SEM imaging can be used to obtain three-dimensional shape and composition information of particles (van Poppel et al. 2005; Adachi et al. 2007; Adler et al. 2013; Conny 2013).

Particle shape is also important for the measurement of specific surface area (SSA), which impacts aerosol reactivity and ice nucleation activity (Brantley and Mellot 2000; Hoose and Möhler 2012). There have been multiple strategies used to obtain particle surface area, including scanning probe microscopy and gas adsorption. AFM has been used to study minerals, but it is time consuming to analyze a sufficient number of particles with this technique and it can miss some of the interior detail (Bickmore et al. 2002; Metz et al. 2005). The major
method used for minerals is the measurement of Brunauer-Emmett-Teller (BET) surface area, where the adsorption of a nonreactive gas to the surface of the particles is used to determine the surface area (Brunauer et al. 1938; Madsen 1977; van Olphen and Fripiat 1979; Prueitt and Webb 1993; Bereznietskii et al. 1998; Schulteifield et al. 2007; Steudel et al. 2009; Sanders et al. 2010; Broadley et al. 2012). BET measurements can show variability for particles depending on the gas used with up to a 37% difference shown between N2 and Kr adsorption (Brantley and Mellott 2000). For standard materials, differences from the same sample are observed from 0.19% to −12.77% for a high SSA material (Hackley and Stefaniak 2013). New methods for the measurement of geometric surface area may provide a lower bound for the surface area of minerals.

In our study, we demonstrate a method for calculating the dimensions of mineral dust particles in the submicron regime using SEM. Previously, SEM has generally been used to look at particles larger than a micron and for elemental identification with energy dispersive X-ray spectroscopy (Laird 2001; Krueger et al. 2003; Clayton and Pearce 2007; Chen et al. 2013). As described above, previous studies using TEM have required lengthy sample preparation techniques and additional assumptions to ascertain the height of particles. While AFM studies have been shown to give accurate detail of the particles, these methods are time consuming for the collection of information on hundreds of single particles. In contrast, high resolution SEM requires no sample preparation and large numbers of particles can be analyzed quickly. We demonstrate our method on a variety of commercially available minerals.

**EXPERIMENTAL METHODS**

Four types of clay minerals used were from the Source Clays Repository of the Clay Mineral Society: low-defect kaolinite, Washington County Georgia, USA (KGa-1b); high-defect kaolinite, Warren County Georgia, USA (KGa-2); montmorillonite, Gonzales County TX, USA (STx-1b); and Na-rich montmorillonite, Crook County WY, USA (SWy-2). The remaining minerals were: NX-Illite (Arginotec, NX Nano-powder, B+M Notenkämper, Munich, Germany), calcium carbonate (>99.95%, Macron Chemicals), and silicon dioxide (quartz; >99%, Sigma Aldrich). Because the calcium carbonate and silicon dioxide were obtained from chemical companies, their physical and chemical properties may differ from geological sources. NX-Illite is a mixture of minerals that has been suggested to have a similar composition as mineral dust that undergoes long-range atmospheric transport (Broadley et al. 2012). In this study, we have investigated aerosolized samples, which may have a different composition than the bulk samples.

Aerosol particles were generated by directing a stream of nitrogen at 1.5 lpm toward the mineral dust while agitating it. The entrained dust particles were directed into a cascade impactor (PIXE International Corp., Tallahassee, FL, USA) backed with a pump at 1.0 lpm. Particles were impacted near the edge of the silicon wafer (Virginia Semiconductor Inc., Fredericksburg, VA, USA) for SEM analysis. For quartz particles, the silicon substrate was coated with a thin layer of Formvar 15/95 (Electron Microscopy Science, Hatfield, PA, USA) to prevent damage to the substrate from the particle impaction.

A FEI NanoSEM 630 FESEM operated at 3 keV was used to image the particles. Under these conditions, a resolution of <2 nm can be obtained. Particles were imaged in top-down and side-on configurations. Top-down refers to the standard SEM configuration. To image side-on, the substrates were placed in a perpendicular orientation at an angle of 5° with respect to normal, and a tilt correction was used to remove distortion. The particles were imaged at an orientation that allowed for the greatest emission of secondary electrons toward the detector, which results in the best resolution. Top-down and side-on images were analyzed to measure the aspect ratios using ImageJ (National Institutes of Health, Bethesda, MD, USA). In the top-down orientation, the aspect ratio is calculated from the ratio of the length to the width of the particle. The length is defined as the longest distance across the center of the particle; the width is measured across the longest distance in the orthogonal direction. In the side-on orientation, the aspect ratio is calculated as the ratio of the width to the height of the particle. In this study, we have investigated minerals that can be described by these two aspect ratios. We note, however, that some types of minerals have shapes that are best described by three aspect ratios (e.g., rectangular prisms). For these types of particles, the aspect ratio in the top-down orientation will not be close to unity. In this case, two aspect ratios will be averaged together in the side-on orientation. Using the top-down orientation, however, the length and width of the particles is measured, and the height is measured in the side-on orientation. By combining the top-down length and width measurements and the side-on measurement of height, the three aspect ratios can be determined. Only particles with a dimension less than 1 μm were analyzed due to large quartz impurities present in some of the clay mineral samples (Clayton and Pearce 2007). Using the top view of the particles, 198 to 511 particles were analyzed for each sample. Due to the low depth of field with the SEM for the side view, 90 to 167 particles were analyzed for each sample.

The geometric SSA of the particles is defined as area per unit mass. To calculate the particle surface area, we have assumed three geometries for the particles: hexagonal prisms, rectangular prisms, and spheroids. Using the top-down SEM image, the length and width of each particle is measured. The aspect ratio for the side-on orientation is used to obtain the height of the particle. Using the geometry of interest, the ratio of the surface area to volume is calculated for each particle. The volumes are then normalized by the density of the particles to give units of m2/g. The densities in units of g/cm3...
used for the calculations were: 2.71 for calcite, 2.65 for quartz, 2.8 for illite, 2.65 for kaolinite, and 2.5 for montmorillonite (Haynes 2013). The SSAs for each particle are summed and divided by the number of particles to give an average SSA.

RESULTS AND DISCUSSION

To demonstrate the use of SEM for the measurement of the aspect ratios of mineral particles, we use commercially available samples of calcite, quartz, illite, kaolinite, and montmorillonite, as described above. We report the values derived for these commonly used samples, but this technique can be extended to any sample of interest. In Figures 1–3, we show histograms of the aspect ratios for each type of mineral in the top-down and side-on orientations with representative SEM images. Figure 4 shows a box-and-whisker plot of the aspect ratios of all particle types measured for both orientations. The central value shown is the mean, the box indicates the 25th and 75th percentile, and the whiskers mark the 5th and 95th percentiles. Table 1 summarizes the mean aspect ratios obtained with standard deviations. All of the particles have aspect ratios of approximately unity when only the top-down orientation is measured. The major difference between particle dimensions arises from the side-on view of the particles. There is no dependence of the aspect ratio on the size of the particles for the submicron diameters reported in this study. In the supermicron regime, a size dependence is present for some samples, such as the montmorillonite SWy-2, due to mineral contaminants. While both kaolinite samples are >96% pure, the STx-1b contains 67% montmorillonite and up to 3% quartz. The SWy-2 bulk sample contains 75% montmorillonite, 8% quartz, and 16% feldspar while the <2 micron fraction contains 95% montmorillonite and 4% quartz (Chipera and Bish 2001). Plots of the aspect ratio as a function of particle size for submicron and supermicron particles are provided in the online supplemental information. We note that the described SEM technique can be extended without alteration to study supermicron particles.

The top-down and side-on orientations of calcite have similar aspect ratios of 1.29 ± 0.20 and 1.27 ± 0.20, respectively (Figures 1a–c, Table 1). The range of aspect ratios observed is shown in Figure 1c, where the peaks that represent the measured aspect ratios for top-down and side-on orientations match closely. Figures 1a and b show SEM images that illustrate that the particles have a near unity aspect ratio in both orientations. Note that surface roughness affects the aspect ratios of these particles, which accounts for the aspect ratios being slightly greater than one.

Quartz has a higher aspect ratio than calcite in the top-down orientation of 1.38 ± 0.28, and deviates more from calcite with its side-on aspect ratio of 1.64 ± 0.49 (Figures 1d–f, Table 1). These aspect ratios indicate that quartz is slightly...
elongated, while calcite is approximately spherical, in agreement with the literature (Siegesmund et al. 2002). Figure 1f shows that the distribution of the aspect ratios for quartz in the side-on orientation peaks at a higher value than for calcite. From the top-down and side-on SEM images of quartz, the surface of these particles is smooth in contrast to the roughness of the calcite particles (Figures 1d and e).

The aspect ratios of the clay minerals in the top-down orientation range from 1.35 to 1.44, in agreement with literature values of approximately 1.4 (Table 2; Robertson et al. 1954; Nadeau 1985; Becket et al. 1997). These values are larger than the aspect ratio of calcite in the top-down orientation at 1.29 ± 0.20, but are similar to that of quartz at 1.38 ± 0.28 (Table 1). This result indicates that when looking at the particles in the top-down orientation in SEM or TEM, the particles look approximately spherical or cubic (Figures 2a and d, 3a, d and g). The range of aspect ratio values for the top-down orientation is similar for all clay minerals used in this study (Figures 2c and f, 3c, f, and i).

The aspect ratios measured for the side-on orientation of the clay mineral dust are significantly different than the other minerals. The two kaolinite samples studied (KGa-1b and KGa-2) have similar aspect ratios in both top-down and side-on orientations and similar particle morphologies (Figure 2). The distribution of aspect ratios is wide, but similar for the two types of kaolinite (Figures 2c and f). The particles have regular polygonal structures with sharp, defined edges and flat surfaces (Figures 2a, b, d, and e). Literature values for the aspect ratio of kaolinite measured with TEM report median values of approximately 10 with a minimum of 2.8 and a maximum of 17.67 (Table 2; Robertson et al. 1954; Jepson and Rowse 1975; Nadeau 1987; Becket et al. 1997). The kaolinite aspect ratios obtained in this study have values near the median of the literature results.

Of the clay minerals, the lowest aspect ratio is measured for the NX-illite sample at 4.80 ± 2.23 (Table 1). The distribution of aspect ratios of the NX-illite particles is wide when compared to calcite or quartz, but smaller than the spread for the aspect ratios of kaolinite and montmorillonite (Figure 3c). NX-Illite samples vary in composition, but are greater than 60% illite with additional contributions from illite-smectite mixed layers, feldspar, kaolinite, quartz, and carbonates (Broadley et al. 2012). As a result, the SEM images show different types of particles, rather than just illite (Figures 3a and b). The measured aspect ratio is an average over this diverse composition, which could result in a lower aspect ratio compared to the other clay minerals in this study. TEM studies have reported aspect ratios for illite from 2.10 to 120 (Table 2; Nadeau 1985; Inoue and Kitagawa 1994; Becket et al. 1997). From the SEM images, the particles are irregular and feathery in morphology (Figures 3a and b).

The montmorillonite particles (STx-1b and SWy-2) have the largest range of aspect ratios among the clay minerals (Figures 3f and i) even though they have similar average
aspect ratios to kaolinite (Table 1). They also have the largest variation in particle morphology. STx-1b particles have a wispy/flakey appearance with rough edges (Figures 3d and e). SWy-2 has structures with flat regular features, but there are

**TABLE 1**

Average aspect ratios and standard deviations for the minerals used in this study for both the top-down and side-on orientations

| Species           | Aspect ratio top | Aspect ratio side |
|-------------------|------------------|-------------------|
| Calcite           | 1.29 ± 0.20      | 1.27 ± 0.20       |
| Quartz            | 1.38 ± 0.28      | 1.64 ± 0.49       |
| NX-Illite         | 1.44 ± 0.32      | 4.80 ± 2.23       |
| Kaolinite KGa-1b  | 1.35 ± 0.22      | 7.98 ± 3.94       |
| Kaolinite KGa-2   | 1.39 ± 0.27      | 8.89 ± 3.72       |
| Montmorillonite STx-1b | 1.38 ± 0.25 | 7.49 ± 4.20 |
| Montmorillonite SWy-2 | 1.44 ± 0.29 | 9.14 ± 4.67 |
also particles similar in morphology to STx-1b and NX-illite. The SWy-2 structures were closer in morphology to the kao-
linite samples than the STx-1b, but did not have the regular
hexagonal structure of kaolinite (Figures 3g and h). In our
experimental measurements, the kaolinite and montmorillonite
particles have similar mean aspect ratios in both the top-down
and side-on orientations, but the montmorillonite particles
have a much greater range of measured values (Figure 4).
This range of measurements is reflected in the larger standard
deviation of the montmorillonite aspect ratios in comparison
to kaolinite (Table 1). Previous studies of montmorillonite
have had a much larger range of aspect ratios than kaolinite
from 3.10 to 448 (Table 2; Nadeau 1985; Becket et al. 1997).

SSA was calculated from the SEM images of our aero-
solized samples to compare with previously published BET
and AFM measurements of bulk samples. As stated previ-
ously, the composition of the aerosolized, submicron sam-
ple may differ from the bulk composition. The particles
studied have a variety of shapes, and as a result, we have
calculated the SSA for each type of particle studied assum-
ing that the particles are hexagonal prisms, rectangular
prisms, and spheroids. Because BET measurements use a
bulk sample for analysis, the polydispersity of the samples
are included in the analysis as described in the Experimental
Methods. This distribution is then used to calculate the bulk
geometric SSA of the submicron fraction. The SSA of

| Species   | Top | Side | Lit. top | Ref.                  | Lit. side | Ref.                  |
|-----------|-----|------|----------|-----------------------|----------|-----------------------|
| NX-Illite | 1.44| 4.80 | min = 2.10 | Inoue and Kitagawa (1994) | min = 11.3 | Becket et al. (1997) |
| Kaolinite | 1.35–1.39 | 7.98–8.89 | min = 1.29 | Robertson et al. (1954) | min = 2.8 | Becket et al. (1997) |
| Montmorillonite | 1.39–1.44 | 7.49–9.14 | 1.68 | Nadeau (1985) | min = 3.10 | Becket et al. (1997) |

### Table 3

| Species   | SSA hex. prism (m²/g) | SSA rect. prism (m²/g) | SSA spheroid (m²/g) | BET | Ref.                  | AFM | Ref.                  |
|-----------|-----------------------|------------------------|---------------------|-----|-----------------------|-----|-----------------------|
| Calcite   | 8.8                   | 12.0                   | 12.2                |     |                       |     |                       |
| Quartz    | 9.1                   | 11.9                   | 12.2                |     |                       |     |                       |
| NX-illite | 33.5                  | 38.3                   | 45.6                | min = 94 | Steudel et al. (2009) |     |                       |
|           |                       |                        |                     | max = 104.2 | Broadley et al. (2012) |     |                       |
| KGa-1b    | 19.6                  | 21.5                   | 27.4                | min = 10.7 | Schuttlefield et al. (2007) | 10.1 | Bickmore et al. (2002) |
|           |                       |                        |                     | max = 16.1 | Sanders et al. (2010) |     |                       |
| KGa-2     | 28.7                  | 31.1                   | 40.4                | min = 22.4 | Bereznitski et al. (1998) | 24.1 | Bickmore et al. (2002) |
|           |                       |                        |                     | max = 24 | Madsen (1977) |     |                       |
| STx-1b    | 45.1                  | 49.6                   | 62.9                | min = 82.9 | Sanders et al. (2010) |     |                       |
|           |                       |                        |                     | max = 83.8 | van Olphen and Fripiat (1979) |     |                       |
| SWy-2     | 54.5                  | 58.9                   | 77.1                | min = 31.82 | van Olphen and Fripiat (1979) |     |                       |
|           |                       |                        |                     | max = 34.0 | Sanders et al. (2010) |     |                       |
calcite and quartz were calculated and are expected to be close to BET measurements because of their smooth surfaces. When our measurements are compared to the BET measurements for the same species, there is good agreement for the kaolinite species between the SEM data, BET measurements, and AFM measurements (Table 3). We would expect good agreement in this case because of the smooth shape of the particles. The SSA of the SWy-2 montmorillonite sample was overestimated, which can be accounted for by the large difference between bulk composition of SWy-2 and the <2 µm fraction. While the bulk sample contains up to 8% quartz and 16% feldspar, the <2 µm fraction contains up to 4% quartz (Chipera and Bish 2001). Quartz has a smaller SSA than montmorillonite, which will especially effect the calculation of SSA in the supermicron regime. As a result, the bulk SSA reported in the literature will be smaller than our experimental estimate of the submicron fraction. The SSA of NX-illite is underestimated compared with measurements for illite because NX-illite is a mixture of minerals rather than pure illite (Broadley et al. 2012). The SSA of STx-1b is also significantly underestimated because these species are not regular polygons. Instead, they have a wispy/flake structure that results in additional surface area that is accounted for in the BET measurement, but not in the SEM measurement. While BET measurements with N₂ do not penetrate the interlayer spacing of montmorillonite (Grim 1968), they measure surface heterogeneities and pores, and therefore may result in larger values than our calculations. Water in interstitial spacings is removed by heating prior to BET measurements and depleted by the vacuum conditions in the SEM. As a result, both BET and SEM measurements are unlikely to be affected differently by the amount of interstitial water. In summary, our calculations based on the SEM images show that there is good agreement between the SSA measurements found with SEM and BET when the particles are smooth in shape, and that we obtain an underestimate for more complex particle morphologies.

CONCLUSIONS

SEM provides a method of characterizing the aspect ratios of particles in the top-down and side-on orientations with little sample preparation. To demonstrate this technique, we have used common, commercially available components of mineral dust, including calcite, quartz, NX-illite, kaolinite (KGa-1b, KGa-2), and montmorillonite (STx-1b, SWy-2). In the top-down orientation (i.e., the usual SEM orientation), all mineral dust components studied have an aspect ratio of approximately unity. In the side-on orientation, the aspect ratio of calcite is approximately unity, and the aspect ratio of quartz is slightly greater than for the top-down orientation. The clay minerals that we investigated have significantly larger aspect ratios in the side-on orientation than calcite and quartz. NX-IIIIt contains the smallest aspect ratio of the clay minerals and montmorillonite has the largest range of observed values. These aspect ratio measurements were coupled with the size distribution to estimate the geometric SSA of the particles. The results are similar to BET measurements for smooth kaolinite particles, while they underestimate the surface area determined with BET for nonsmooth NX-illite and STx-1b montmorillonite. The SEM method introduced in this article will allow a fast, easy measurement of the aspect ratios of mineral dust samples of interest. As a result, it has the potential to improve the calculation of the optical properties of nonspherical mineral dust particles for climate modeling and remote sensing.

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SUPPLEMENTAL MATERIAL

Supplemental data for this article can be accessed on the publisher’s website.

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