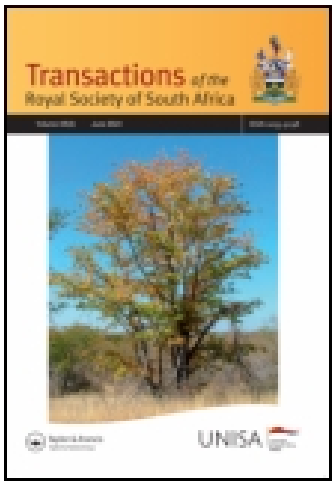

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Sulphur isotopes in the central Namib Desert ecosystem

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The Namib Desert is hyper-arid in terms of rainfall, but its ecology is influenced by frequent fog events. Fog utilisation by Namib biota has been well studied, but its role in nutrient deposition and cycling, particularly with respect to soil processes, still has open questions. Given its potential for distinguishing between various ecosystem components and fluxes, sulphur isotopic composition ($\delta^{34}S$) is evaluated here as a passive tracer of aerosol deposition and plant water sources in the Namib. Measurements of $\delta^{34}S$ in Namib fog, groundwater, soils, plants and aerosols are presented and are consistent with the previously described system of sulphur cycling: primary marine sulphur accumulates as gypsum in the gravel plains and is redistributed by wind. Kuiseb River sediments had a wide range of $\delta^{34}S$ values, with several samples that were quite depleted relative to soils, plants, groundwater and gypsum of the gravel plains. This depleted signal appears more commonly in the fine (0.5, 1.0 μm) rather than in the coarse (1.5, 7.6 μm) aerosol size fractions. Fog and aerosol $\delta^{34}S$ values are consistent with local dust as a major sulphur source, limiting the utility of $\delta^{34}S$ as a unique tracer of fog deposition. It can still provide useful information in certain situations. For example, the 16.5‰ $\delta^{34}S$ value for the brackish groundwater at Hope Mine is distinct from the 10.2‰ value in *Welwitschia mirabilis* stem material at that site. This type of comparison could be one useful line of evidence in evaluating plant water sources.

INTRODUCTION

Although rainfall pulses are important for biogeochemical processes in arid ecosystems (Seely, 1978; Jacobson & Jacobson, 1998; Huxman *et al*., 2004), many organisms in the Namib Desert make use of the much more frequent fog and dew events to survive, and even thrive, between these rare pulses. Decades of research in Namib Desert ecosystems has demonstrated the utilisation of fog water by Namib biota (Louw, 1972; Hamilton & Seely, 1976; Seely & Hamilton, 1976; Loris, 2004; Henschel & Seely, 2008; Schachtschneider & February, 2010; Ebner *et al*., 2011; Warren-Rhodes *et al*., 2013), the general occurrence and characteristics of fog events (Lancaster *et al*., 1984; Pietruszka & Seely, 1985; Olivier, 1995; Henschel *et al*., 1998; Hachfeld & Jurgens, 2000; Eckardt *et al*., 2013), as well as geochemical aspects of the Namib ecosystem relating to fog deposition (Eckardt & Schemenauer, 1998; Goudie & Parker, 1998; Kaseke *et al*., 2012). The total water and nutrient flux due to fog deposition on soil and plants remains uncertain at the landscape scale, although studies have shown appreciable soil wetting (up to 4 cm depth) from direct fog deposition as well as fog-drip onto soil from plant leaves (Gut, 1988; Soderberg, 2010; Ebner *et al*., 2011; Warren-Rhodes *et al*., 2013). Wetting of soil surfaces from dew deposition and vapour adsorption can be significant, and these processes remain confounding factors for quantifying the total water and nutrient flux associated with fog (Kaseke *et al*., 2012; Eckardt *et al*., 2013). Sulphur isotopic composition ($\delta^{34}S$, defined below) can be a useful passive tracer in the environment given the large differences in isotopic composition that can exist in different ecosystem components (e.g. anaerobic sediments, sea salt, sulphide minerals)(Eckardt & Spiro, 1999). Here we assess the utility of $\delta^{34}S$ as a tool for studying ecosystem processes such as deposition of aerosols and for identifying plant water sources in the Namib. Can $\delta^{34}S$ of plant material be used to distinguish between the uptake of groundwater, soil water and fog? The hypothesis is that $\delta^{34}S$ can make this distinction, with fog having a distinctly enriched marine signature. Such an assessment, however, requires an understanding of $\delta^{34}S$ in sulphur sources, fluxes of sulphur among ecosystem compartments, and how the sulphur sources interact with plant water sources such as fog, rain and groundwater. Significant interaction between fog and local dust, for example, could render $\delta^{34}S$ less useful as a unique tracer of fog uptake by plants.

Dust and fog both occur frequently in the Namib, although their interactions are not immediately clear. Elsewhere, dust has been shown to prevent clouds from releasing rain due to the overabundance of condensation nuclei (Rosenfeld *et al*., 2001). Despite frequent strong "berg winds" and impressive but less common dust storm events, the Namib is known for having some of the lowest background aerosol chemical concentrations in the world, e.g. Pb of 0.6 ng/m^3 (Bollhöfer & Rosman, 2000) and S of 200 ng/m^3 (Annegarn *et al*., 1983). A recent global modelling effort lists the background S in <2.5 μm aerosols over Africa as 278 ng/m^3 (Liu *et al*., 2009), which lies between the Namib value and that of a background sampling station in Zimbabwe – 452 ng/m^3 (Nyanganyura *et al*., 2007). Potential sources of sulphur in aerosols include gypsum ($CaSO_4$), which is widespread in the central Namib soils, dimethyl sulfide (DMS) from the ocean, sea-salt, hydrogen sulfide from sulfate-reducing bacteria on the Namibian shelf, regional biomass burning, fossil fuel burning, and other terrestrial materials that are mobilised from dry pans and riverbeds (Annegarn *et al*., 1983; Eltayeb *et al*., 1993; Eckardt &

Spiro, 1999; Eckardt *et al*., 2001a, b). The chemical composition of Namib fog indicates that particles of dust have likely contributed elements such as sulphur and calcium (e.g. from gypsum) by dissolving into the water droplets that make up fog (Eckardt *et al*., 2001b). In this study, we report $\delta^{34}S$ of size-segregated aerosols during and after fog events (7.6, 1.5, 1.0 and 0.5 μm mean aerodynamic diameter), with the hypothesis that coarser size fractions (7.6 and 1.5 μm) have more enriched $\delta^{34}S$ values due to the contribution of either inorganic marine sulphur or gypsum dust compared to the contribution of fine-grained terrestrial materials or organic-derived sulphur (e.g. biomass burning, hydrogen sulphide released from marine sediments) in the finer size fractions (Annegarn *et al*., 1978, 1979, 1983; Eltayeb *et al*., 1993; Eckardt & Spiro, 1999; Posfai *et al*., 2003). These aerosols can each play a role in defining the $\delta^{34}S$ of fog from the point that the water droplets form until they dissipate into dry aerosols or wet soil and plant surfaces.

Using sulphur isotopic composition as a passive environmental tracer requires $\delta^{34}S$ values that are distinct among the end members but sufficiently consistent within each. If $\delta^{34}S$ of plant material is to be considered a marker of fog uptake, the various sulphur sources and uptake pathways need to be assessed, including soil, groundwater, fog and aerosols. We present $\delta^{34}S$ data from soils, plants, aerosols, groundwater and fog from the central Namib Desert collected from 2007 to 2009. These data will contribute to a first order assessment of how well the various end members can be differentiated.

METHODS

Plant (n=74), soil (n=26), water (n=5) and aerosol (n=76) samples were collected along the precipitation gradient in the Central Namib. Sampling sites included dunes, gravel plains and the ephemeral Kuiseb River at various distances from the coast (Figure 1): Rooibank (10 km; -23.167° S, 14.635° E), Swartbank (35 km; -23.322° S, 14.814° E), Gobabeb (56 km; -23.560° S, 15.038° E), Hope Mine (78 km; -23.569° S, 15.264° E), and Ubib (77 km; -23.120° S, 15.186° E). Plant species sampled included common trees that tend to grow in and around the Kuiseb River (*Acacia erioloba*, *Faidherbia albida*, *Tamarix usneoides*); shrubs of the gravel plains (*Welwitschia mirabilis, Zygophyllum stapffii*, *Arthraerua leubnitziae*, *Calicorema capitata*); a shrub (*Trianthema hereroensis*) and grass (*Stipagrostis sabulicola*) that grow on dunes; and a melon-bearing stem succulent (*Acanthosicyos horridus*) which tends to grow in interdune valleys. A gypsum sample was collected from one of the Ubib playas and identified in the field by F. Eckardt. Plant, soil and aerosol samples were dried at 100°C, and plant samples were homogenised in a Wiley Mill prior to analysis. Water samples (groundwater n=3, fog n=1, rain n=1) were prepared by precipitating sulfate from the samples through the addition of barium chloride. Isotopic composition was not quantifiable in several samples due to low sulphur mass or poor combustion – 1 rain (22 June 2009), 2 plant, 12 soil and 17 aerosol samples. Fog was collected on 21 June 2009 using the Standard Fog Collector (Schemenauer & Cereceda, 1994) at Gobabeb, which is oriented to the northwest. No manual pre-rinsing was performed. Fog and light rain had occurred on the previous two days.

Aerosols were collected with a High Volume Cascade Impactor system (Turekian *et al*., 2001, Tisch Environmental Inc., 2004) at flow rates averaging about 1000 l per minute (LPM). Pre-ashed glass fibre (GF) filters on four impactor stages (7.6, 1.5, 1.0 and 0.5 μm mean aerodynamic diameter) were used to collect aerosols for 11 h sampling periods: "Day" (~7 am to 6 pm local time) and "Night" (~7 pm to 6 am) from 21 June to 2 July 2009. See Soderberg (2010) for flow rate and size cutoff calculations. Collection was performed at Gobabeb, 1.5 m above the ground in a location as far as possible (~500 m) from the daily activities of the research station. Filters were pre-ashed at 550°C for 4 h and no sulphur was detected in field or laboratory blanks of the slotted GF filters. The backing filter media (<0.5 μm mean aerodynamic diameter) contained a binder that retained a sulphur blank after ashing, and thus these results are not presented. Immediately following collection, the GF filters were folded in half to protect the collection surfaces, enclosed in pre-ashed aluminium foil, and kept frozen until analysis.

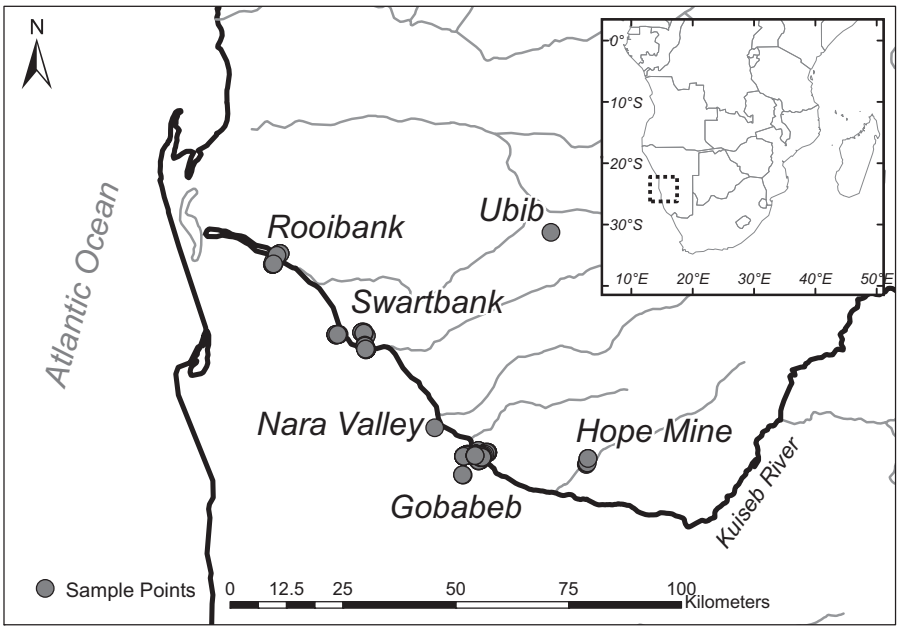

Figure 1. Sampling locations.

Sulphur isotope values were determined using an elemental analyser (EA, Carlo Erba) coupled to an isotope ratio mass spectrometer (VG/Elementar Optima). Isotopic composition is reported in delta notation as a ratio of heavy to light isotope abundance relative to the standard Canyon Diablo Troilite (CDT) (equation 1), and results are shown in "per mil" (×1000) with the symbol "‰" (Coplen, 2011). Lower $\delta^{34}S$ values are referred to as "depleted" and higher values as "enriched", reflecting the relative abundance of the heavier isotope as compared to that of the CDT standard.

$$\delta^{34}S = \left(R_{sample}/R_{CDT}\right) - 1 \quad (1)$$

The isotope value is corrected for contribution from ^{18}O content (Giesemann *et al.*, 1994) after adjusting the value to an internal standard ($BaSO_4$). Reproducibility is typically better than ±0.2‰ with the continuous flow Optima system.

RESULTS

Plant material $\delta^{34}S$ ranged from 0.6‰ in *T. usneoides* in the Kuiseb River at Gobabeb to 21.7‰ in *Z. stapffii* in the gravel plains at Gobabeb. Most values fell between 10 and 17‰ (Figure 2 and Supplemental Information Table S3), with a mean of 13.5 +/- 3.6 ‰ (1 standard deviation). Total sulphur content was significantly lower in stem material than leaf

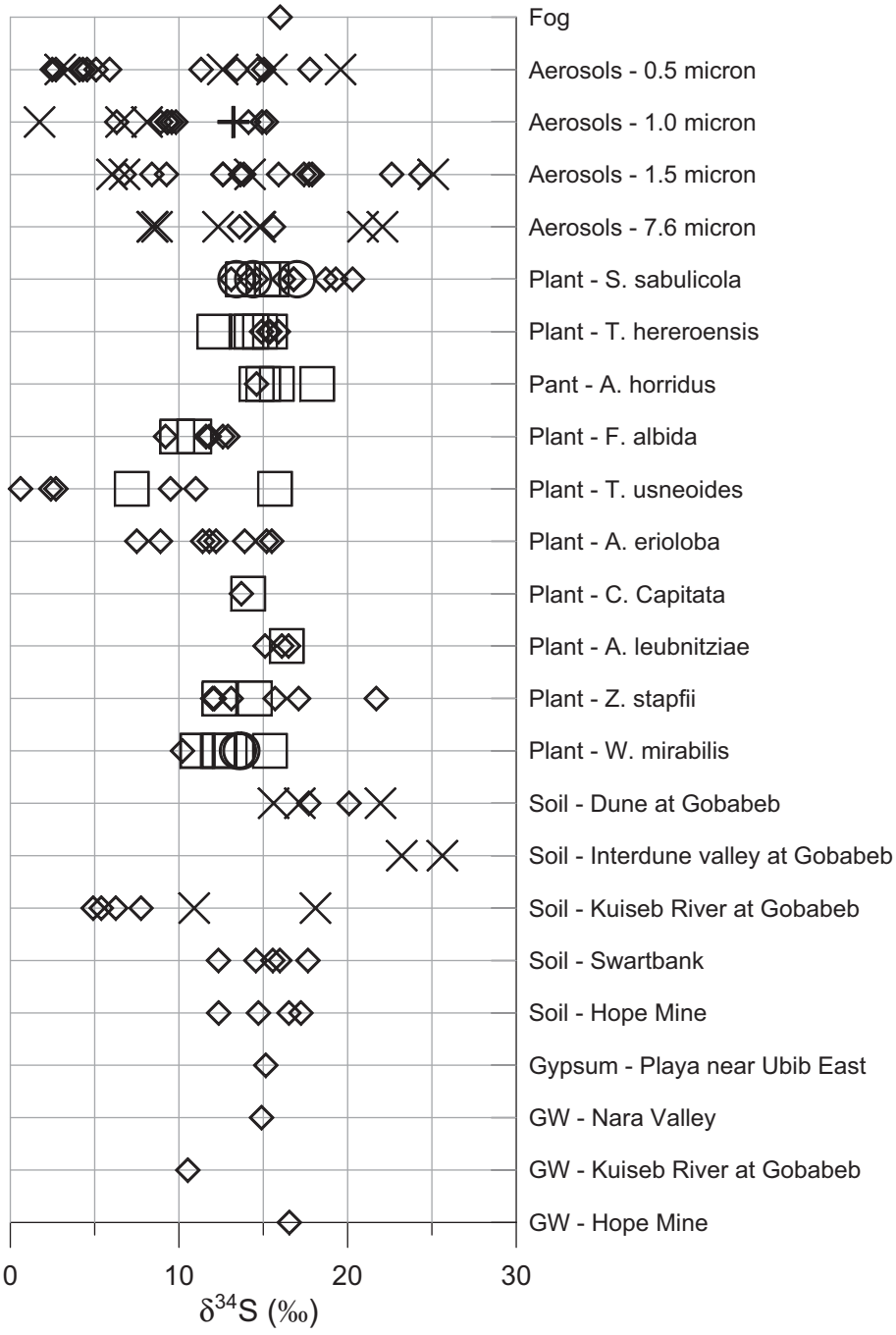

Figure 2. All $\delta^{34}S$ values for water, plant, soil and aerosol samples. For plants, squares are leaf material, circles are lateral root material and diamonds are stem material. Samples with trace amounts of sulphur in the analysed aliquot are shown with an "X" symbol, and the one sample with non-detected sulphur mass is shown with a "**+**" symbol.

material (overall mean values of 1980 mg/kg and 7620 mg/kg, respectively; $p<0.1$ using Student's *t*-test with unequal variances), with extreme values of 21 300 mg/kg and 52 300 mg/kg in leaf samples of *T. usneoides* at Rooibank and *Z. stapfii* at Gobabeb, respectively. Stem and leaf $\delta^{34}S$ values were not significantly different when all species are considered together, although certain species such as *T. usneoides* and *W. mirabilis* did show some differences.

Soil (including dune sand and dry riverbed sediment) $\delta^{34}S$ ranged from 4.9‰ in Kuiseb River soil near a *T. usneoides* individual at Gobabeb to 25.6‰ in the interdune valley between "Station Dune" and "High Dune" at Gobabeb near an *A. horridus* individual. The gypsum sample had a value of 15.2‰, similar to the nearby "Ubib East" gypsum (15.4‰) and spring water (15.6‰) reported by Eckardt and Spiro (1999). Overall, Kuiseb River soils had depleted $\delta^{34}S$ composition (8.9 +/- 5.0‰) relative to the gravel plains (15.2 +/- 2.2‰), inselberg (15.2 +/- 2.0‰), dune (18.5 +/- 2.5‰) and interdune (23.2 and 25.6‰) soils. The Kuiseb soils were taken from two locations at Gobabeb – four samples near a *T. usneoides* individual (4.9 to 7.8‰) and two samples near an *A. erioloba* individual (10.9 and 18.1‰). The four depleted samples include both surface and subsurface samples down to 12 cm depth. Several of the dune, interdune and riverbed samples had trace levels of total sulphur (less than 3.4 μg in the aliquot analysed, or 50 to 100 mg/kg depending on the mass of the aliquot; see Supplemental Information Table S2). The isotope ratio measurement is more sensitive than the elemental content measurement, allowing for $\delta^{34}S$ to be quantified for the low sulphur samples.

Groundwater from the alluvial aquifer at Gobabeb had a depleted $\delta^{34}S$ composition of 10.5‰, which is the same value reported previously for this borehole (Eckardt & Spiro, 1999). Groundwater at Hope Mine was approximately 25 m below the ground surface (accessed via exploratory boreholes with no casing below 1–2 m), and had $\delta^{34}S$ of 16.5‰ and a total salinity content of 28 (measured with a handheld optical instrument in Practical Salinity Units, approximately 28 000 mg/kg total dissolved solids). A groundwater sample from Zebra Pan, approximately 25 km northeast from Hope Mine was reported to have a $\delta^{34}S$ of 15.1‰ with SO_4 and Cl dissolved ion content of 14.2 and 29.1 meq/L, respectively (Eckardt & Spiro, 1999), implying a much lower salinity than the Hope Mine groundwater.

The coarse aerosols had a mean $\delta^{34}S$ of 14.3 +/- 5.6‰ and were significantly enriched relative to the fine aerosols (0.5 and 1.0 μm) with a mean $\delta^{34}S$ of 9.6 +/- 5.1‰ ($p<0.01$). It remains possible that a different composition existed in the ultra-fine (<0.5 μm) fraction, which was not included in this analysis due to a high sulphur blank in the backing filter media. The integrated signal observed in the fog water sample (16.0‰) was closer to the coarse than the fine aerosol values. Air parcel back-trajectory analysis was performed using the HYSPLIT model (Draxler & Rolph, 2011) to confirm the direction of potential fog and aerosol sources suggested by local wind direction data (Soderberg, 2010).The first three days of aerosol sampling saw two fog events (21 and 23 June 2009) and one afternoon light rain event (22 June) which came from the west (i.e. the same Atlantic Ocean water source as the fog). From 24 June to 2 July a strong East Wind brought hot dry conditions. No distinctive difference was found in either the coarse or fine aerosols on foggy days compared to East Wind days (Figure 3).

DISCUSSION

The origins and cycling of the sulphur accumulations in the gravel plains, particularly with respect to the formation and aeolian redistribution of gypsum have been previously described (Eckardt & Spiro, 1999; Eckardt *et al*., 2001a). The most recent interpretation is that the widespread gypsum sulphur in the central Namib results from a dynamic mechanism involving marine aerosol deposition, primary gypsum formation in salt pans where the marine sulphur interacts with terrestrial calcium, and deflation of the pans leading to redistribution (Eckardt *et al*., 2001a). Here we have shown that the same general isotopic composition of the gypsum deposits is seen in plants, soils and groundwater of the gravel plains. Some enrichment was observed in dune and interdune plants and soils around Gobabeb, indicating less influence of gravel plain sulphur and potentially more primary marine deposition. The Kuiseb alluvial groundwater at Gobabeb has a somewhat depleted isotopic composition, likely due to its interaction with aquifer materials and sediments within its catchment, and this signal is reflected in the plant material of Kuiseb trees. *Welwitschia mirabilis* stem material was similarly depleted, although its lateral roots and leaf tips ranged up to the enriched composition of the surrounding soils. It should be noted that although the Hope Mine groundwater and soil results presented here from samples immediately adjacent to the sampled *W. mirabilis* individual had enriched isotopic compositions, Eckardt and Spiro (1999) found depleted $\delta^{34}S$ in gypsum (3.1‰) and sulphide (8.8, 8.5‰) samples nearby. Kuiseb sediments at Gobabeb exhibited a wide range of $\delta^{34}S$ including a significantly depleted composition in sediments close to a *T. usneoides* individual that had higher $\delta^{34}S$ in its leaves and lower $\delta^{34}S$ in its stem. Isotope fractionation within an individual plant remains to be investigated as an explanation for some of the variability observed. *T. usneoides* is a good candidate for this research, as is *A. leubnitziae* given its apparent osmotic control of plant water movement.

One strategy for coping with very dry soils is to increase the solute concentration in the leaves, thereby creating a gradient in osmotic potential that promotes the movement of water from the stems to the leaves (Tissue *et al*., 1991). This strategy is suggested in several of the plants studied here through the difference in total sulphur concentration between stems and leaves. The mean ratio of leaf to stem total sulphur is 3, 4 and 50 in *T. hereroensis, A. horridus* and *Z. stapffii*, respectively. A ratio of 2.8 was also seen in *T. usneoides*, a tree which is known to exude salts from its leaves leaving a greyish appearance. The total sulphur ratio in the two stem succulents *T. hereroensis* and *Z. stapffii* roughly follows their respective ratios of leaf to stem water content: 1.6 and 2.5. However, *A. leubnitziae* reverses this pattern, with a leaf to stem water content ratio of 0.97 and a sulphur content ratio of 0.34. These ratios are consistent with *A. leubnitziae* using osmotic potential to store water in its stem – a water-saving measure that has been observed in the Namib (Jurgens *et al*., 1997).

The fog isotopic composition reported here (16.0‰) was similar to a fog sample value (14.7‰) that was previously reported (Eckardt & Spiro, 1999). Neither of these fog samples was collected under ultra-clean conditions. However, when these data are considered alongside the previously published work relating the dissolved sulphur in Namib fog collected under ultra-clean conditions to gypsum dissolution (Eckardt & Schemenauer, 1998), the working hypothesis should remain that local dust is the dominant sulphur source in Namib fog.

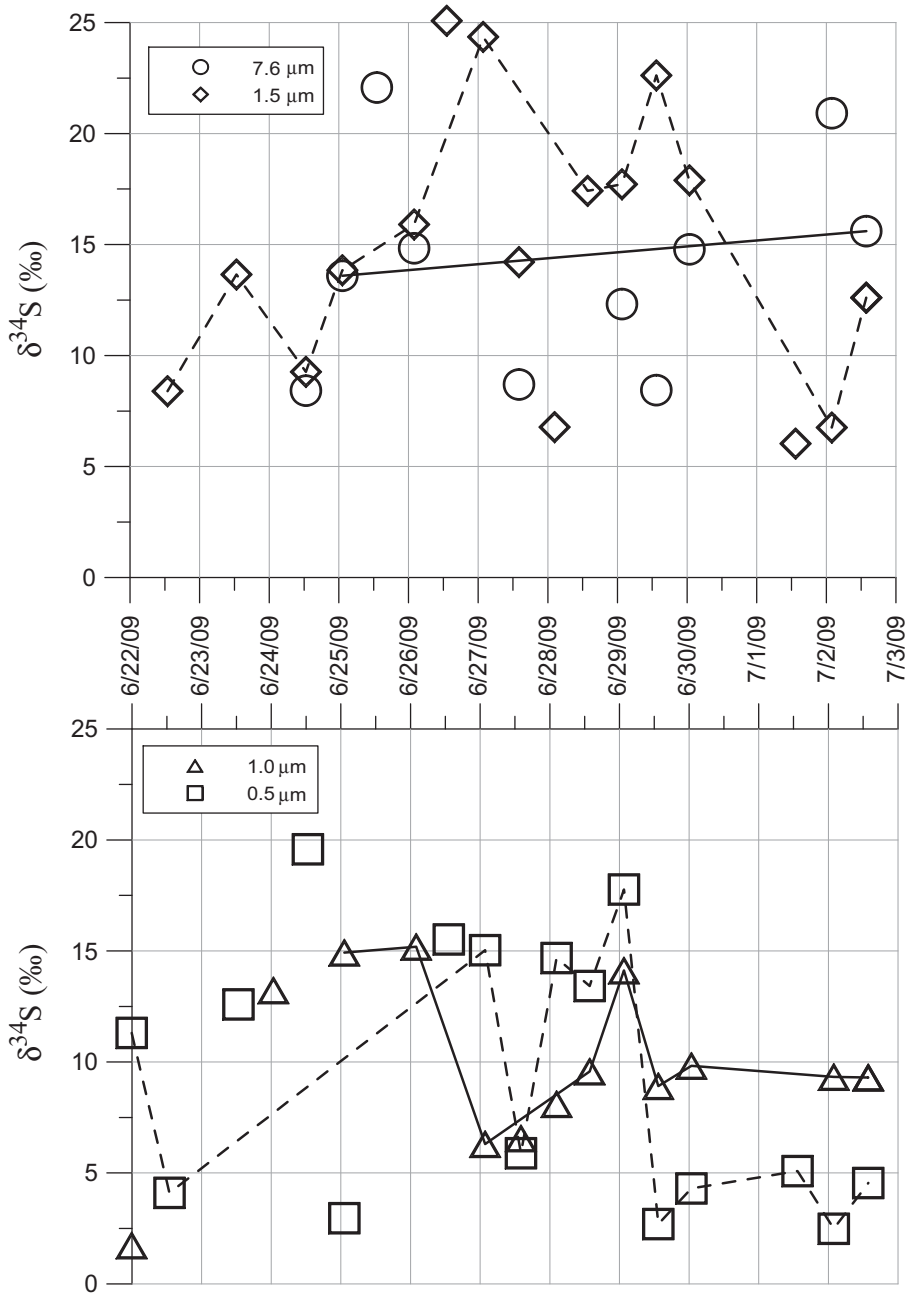

Figure 3. Aerosol $\delta^{34}S$ values for each size fraction over time. Lines connect samples with detectable sulphur mass (i.e. more than trace amounts of sulphur in the analysed aliquot).

The observed aerosol $\delta^{34}S$ values cover several possible end members, including sea-salt (20‰), dimethyl sulfide (18‰), regional gypsum (13.0 to 18.8‰), localised gypsum associated with sulfide deposits (3.1 and 3.4‰), and other terrestrial materials (e.g. local precambrian sulfide-containing rocks at -4.1 to 13.8‰) (Eckardt & Spiro, 1999). The coarse aerosols (1.5 and 7.6 μm) tended to have $\delta^{34}S$ values within the regional gypsum range. Although all size fractions had some samples with $\delta^{34}S$ depleted relative to regional gypsum, this depleted signal was most common in the fine fraction. The Kuiseb soils also exhibited this depleted signal and were the only soils sampled to have $\delta^{34}S$ values in this range. These observations are consistent with local dust as a major source for sulphur in the aerosol size fractions sampled (0.5 to 7.6 μm), but are inconclusive with respect to the ultra-fine aerosols. Mixing of highly depleted H_2S released from marine sediments with enriched dimethyl sulfide released from phytoplankton activity could also produce the somewhat depleted values observed in the fine aerosols. Releases of H_2S from the muddy Benguela sediments are intermittent (Dubecke & Bruchert, 2004; Emeis *et al.*, 2004; Weeks *et al.*, 2004; van der Plas *et al.*, 2007), and thus would be an inconsistent source relative to dimethyl sulfide and gypsum. The hypothesis that the coarser size fractions would have a more enriched $\delta^{34}S$ signal than the finer size fractions is supported by the data presented here. The enriched signal can be explained by gypsum dust, but could also have some contribution from enriched marine sulphur given the similar $\delta^{34}S$ values of these end members and the variability observed in the aerosols. Such a marine contribution could help explain the enriched values in the dune soils, as well as in the 5 coarse aerosol samples that had anomalously enriched $\delta^{34}S$ values (Figure 2).

The depleted signal – present in both coarse and fine aerosols, but more prevalent in the finer size fractions – could generally be explained by contributions from the Kuiseb soils close to the aerosol sampling location. There were, however, 4 samples of fine aerosols (out of 29 quantitated samples for these fractions) with $\delta^{34}S$ values of 1.7 to 3.0‰, which lie between the lowest soil sample value (4.9‰) and the lowest stem values of *T. usneoides* (0.6 to 2.7‰). Part of the explanation for this depleted signal could be the difference between bulk soil samples analysed here and the fraction of the soil that could be part of local dust (Eltayeb *et al*., 1993).

CONCLUSIONS

Overall, sulphur isotopes are of limited utility for tracking fog input to the Namib ecosystem given the apparent dominance of the sulphur signal from the gravel plains. However, sulphur isotopes can help to isolate or eliminate sources or routes of uptake in certain situations. For example, given the large amount of sulphur in the groundwater at Hope Mine along with its enriched isotopic composition, the depleted sulphur isotope composition in *W. mirabilis* plant material is one line of evidence indicating that the groundwater is not being accessed by *W. mirabilis* in this location. Changes to the sulphur isotopic composition within a plant must be considered – *T. usneoides* and *Z. stapffii* appear to be good candidates for investigating fractionation mechanisms associated with, for example, guttation or osmotic control of plant water movement.

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SUPPLEMENTAL DATA

Supplemental data can be found here: http://dx.doi.org/10.1080/0035919X.2014.976778

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