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Phytoliths in particulate matter released by wind erosion on arable land in La Pampa, Argentina

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Silicon (Si) is considered a beneficial element in plant nutrition, but its importance on ecosystems goes far beyond that. Various forms of silicon are found in soils, of which the phytogenic pool plays a decisive role due to its good availability. This Si returns to the soil through the decomposition of plant residues, where they then participate in the further cycle as biogenic amorphous silica (bASi) or so-called phytoliths. These have a high affinity for water, so that the water holding capacity and water availability of soils can be increased even by small amounts of ASi. Agricultural land is a considerable global dust source, and dust samples from arable land have shown in cloud formation experiments a several times higher ice nucleation activity than pure mineral dust. Here, particle sizes in the particulate matter fractions (PM) are important, which can travel long distances and reach high altitudes in the atmosphere. Based on this, the research question was whether phytoliths could be detected in PM samples from wind erosion events, what are the main particle sizes of phytoliths and whether an initial quantification was possible.

Measurements of PM concentrations were carried out at a wind erosion measuring field in the province La Pampa, Argentina. PM were sampled during five erosion events with Environmental Dust Monitors (EDM). After counting and classifying all particles with diameters between 0.3 and 32 µm in the EDMs, they are collected on filters. The filters were analyzed by Scanning Electron Microscopy and Energy Dispersive X-Ray analysis (SEM-EDX) to investigate single or ensembles of particles regarding composition and possible origins.

The analyses showed up to 8.3 per cent being phytoliths in the emitted dust and up to 25 per cent of organic origin. Particles of organic origin are mostly in the coarse dust fraction, whereas phytoliths are predominately transported in the finer dust fractions. Since phytoliths are both an important source of Si as a plant nutrient and are also involved in soil C fixation, their losses from arable land *via* dust emissions should be considered and its specific influence on atmospheric processes should be studied in detail in the future.

KEYWORDS

dust, dust composition, particulate matter, scanning electron microscope (SEM) analysis, phytolith

Introduction

Wind erosion is an important process contributing to soil formation and soil degradation in arid and semi-arid environments. Related dust emissions cause the loss of the most valuable parts of a soil, which not only reduce soil fertility but also influence physical and chemical processes in the atmosphere or remote deposition regions. About 2000 Mt of dust are emitted each year into the atmosphere, of which ¾ are deposited on land surfaces and ¼ on the oceans (Shao et al., 2011). Dust depositions may have an important role in the nutrient cycle of natural ecosystems or low input agriculture as shown from the Amazonas forests (Swap et al., 1992; Kaufmann et al., 2005; Koren et al., 2006) to the Sudan-Sahelian Zone (Jahn 1995; Stahr et al., 1996; Goudie and Middleton 2001). Dust depositions on the oceans activate the phytoplankton growth, which has a direct impact on the global carbon cycle and carbon sequestration as well, as stated by the "Iron hypothesis" of Martin and Fitzwater 1988 and the "Silica hypothesis" of Harrison 2000. Consequently, there is a close relationship between the cycles of dust (D-cycle), carbon (C-cycle) and energy (E-cycle) in the global context (Shao et al., 2011). In longer time-scales, dust has also modifying influences on the climate (Martínez-Garzia et al., 2011).

The sorting processes during a wind erosion event on agriculturally used soils are noticed most clearly in their physical effects, but have also consequences on the chemical composition of the dust and the depositions at the field site. Size, shape and density of single particles or aggregates are the determining criteria for their spread in the atmosphere and finally their transport distances. The contents of soil organic carbon (SOC) and nutrients in the dust fraction have been found to be many times greater than in the original soil. Iturri et al. (2017) found enrichment factors of about five for SOC in the PM_{10} fraction of the eroded material and Webb et al. (2012) reported factors up to seven for Australian dust. Also the nutrient contents of dust are greater with significant higher available P, organic matter and clay contents (Ramsperger et al., 1998; Iturri et al. 2021). Buschiazzo et al. (2007) have measured enrichment factors of two–five for nitrogen and of 1.5–eight for phosphorus for dust transported in 0.13 and 1.5 m height respectively. Measurements in Niger by Sterk et al. (1996) showed 17-times higher contents of K, C, N and P of the dust, trapped in a height of 2 m, than of the topsoil. Nerger et al. (2017) showed a general relationship between the measurement height and the enrichment ratio by summarizing results of different authors. Due to the low net primary production of most regions affected by wind erosion, removed nutrients and carbon can be regarded as irretrievable loss at the eroded site (Yan et al., 2005). Even at the landscape scale these losses are not balanced, because the finer and lighter fractions are transported over long distances and the depositions are spread at much larger areas than the eroded sites.

In addition to the well-studied macronutrients, more of the so-called beneficial elements have recently become into the focus of investigation. One of these elements is silicon (Si), which has a yield-stabilizing effect by increasing resistance to abiotic (drought, nutrient deficiency, metal toxicities) and biotic (fungal diseases, parasite infestation) stress factors, as well as providing culm stability in cereals and other grasses (Epstein, 1999). On a global scale, the Si budget affects the carbon cycle because CO_2 is consumed during silicate weathering (Amelung et al., 2018; Qi et al., 2020). Si is available for plants only as monomeric orthosilicic acid ($Si(OH_4)$), which is obtainable in soils from phytoliths. Phytoliths are amorphous silica particles embedded in plant tissues (biominerals), that remain in soils after decomposition of died plants or fallen leaves (Fernández Honaine et al., 2006). In soils, phytoliths may be found either within individual plant fragments or in the soil mass. The phytolith content varies in the soil horizons between 0.01 and 3% (Schaller et al., 2021) and is increased in the topsoil (Kaczorek et al., 2018). The size of the phytoliths varies from a few 100 nm up to 1 mm. The greatest importance of the phytoliths found in the soils seems to be attributed to the smallest phytogenic structures ($d < 5\,\mu m$). So, they are exactly in a size that makes them susceptible to become part of the suspended dust fraction once they are released from a soil by wind erosion. Latorre et al. (2012) described as first airborne phytoliths in dust in Argentina, whose concentrations are related to agricultural activities and dry weather conditions. The months with higher phytolith concentrations in the air coincided with periods when the soils were not covered with vegetation or when the soils were prepared for sowing. The aeolian release of phytoliths by agricultural activities or by wind erosion would not only reduce the silica content in the soil, but also cause possible undesirable inputs into other biotopes (e.g. eutrophication in water bodies, fertilization of the oceans). Even a small change in the silica content of a few percent would have considerable effects on the soil water holding capacity and so the plant-available water finally (Schaller et al., 2021). Thus, Si - losses in the form of phytoliths also represent a loss of soil quality that is worth investigating in more detail.

On the other hand, it has been demonstrated that dust from arable land has a much higher ice nucleation activity in the atmosphere compared to mineral dust (Conen and Leifeld 2014; Steinke et al., 2016). What causes this higher activity in detail is not yet clear, since soil dust consists of a large number of components (Steinke et al. 2020). The high water absorption capacity of amorphous silica, to which the phytolites belong, could be a first hint that they might be involved in these processes.

Nevertheless, dust is a mixture of single or composed particles different in size, shape and density, and mineral or organic origin. Available measurement methods either examine a small portion of the airflow very accurately, or collect larger amounts of dust without examining it. Optical particle counters use light scattering to detect single particles passing a laser beam and count and classify all particles over certain ranges. They cannot be used to distinguish other important properties as the origin (mineral or organic), the composition (single or composed) or the huge variety of different shapes. Another disadvantage of event-based dust measurements is the often very small quantities that can be collected despite relatively high concentrations in the air. This means that methods that separate certain components from the total load cannot be used and the sample must be analyzed in its entirety. Scanning electron microscopy (SEM) can help to provide additional information about composition and properties of dust particles from samples of low quantities (Hinz et al., 1998; Edgerton et al., 2009; Tolis et al., 2014). Most studies were focused on specific components or elements in the dust, as soot, heavy metals or the mineral and microbiological composition for air quality reasons or source identification (Twiss et al. 1969; Falkovich et al., 2001; Tervahattu et al., 2004; Ramirez-Leal et al., 2014; Rout et al., 2014). Although phytoliths show a great variety of forms and surface structures, nomenclatures are available for their determination, in which they are described in detail (Gallego and Distel 2004; ICPT 2019), so that an identification by SEM should be possible.

Based on these already proven or suspected effects of phytoliths, the aim of our investigations was whether phytoliths could be detected in PM samples from wind erosion measurements at a field site, what are the main particle sizes of phytoliths and whether an initial quantification was possible, at least in a relative way.

Materials and methods

Site description

Wind, wind erosion and dust emissions were measured on a measuring field at the experimental Station of the National Institute for Agricultural Technology (INTA) in Anguil (63.9885°W and 36.577°S) in the Northeastern part of the province La Pampa. The measuring field was orientated along the main wind directions (N—S and S—N) with a size of 1.44 ha (240 × 60 m) and lies within other agricultural land. It was surrounded by pastureland in its immediate vicinity, so a direct input of saltating soil material from the adjacent fields could be excluded. Before the measuring campaign the field was tilled with a disc harrow to remove all vegetation cover. We made sure that the initial conditions at the beginning of the measurements were comparable. Tillage was done three times, before the measurements in August, in September and in November/December. The same tillage tool each time produced comparable roughness. The meteorological conditions led to a rapid drying of the soil surface after 1–2 days each time, so that the initial conditions of the measurements within this time span were similar.

The soils in this region developed from aeolian deposits of Holocene origin and are classified as Typic Ustipsamment with a sand content of 76%, a silt content of 17%, a clay content of 7%, and a carbon content of about 1.5%. The texture class is loamy sand resulting in a medium to high susceptibility to wind erosion. Despite the fairly balanced susceptibility to wind erosion derived from the soil data, there were a strong spatial differentiation in the transport processes due to low terrain structures, so that areas of soil erosion and deposition could be detected at the field immediately adjacent to each other (Siegmund et al., 2018).

Four Environmental Dust Monitors (EDM) were used to measure dust concentrations at heights of 1 and 4 m at two positions, two EDM164 and two EDM107 (GRIMM Aerosol Technique GmbH) (Figure 1). The EDM164 include All-in-One-weather stations (WS500-UMB, Lufft Mess-und Regeltechnik GmbH) to measure simultaneously wind velocity, wind direction, air temperature, air pressure and air humidity. Both positions were placed 40 m away from the field boundary on the longitudinal centerline, so we could define always one position representative for the incoming air and the other one for the outgoing air (Siegmund et al., 2022). The EDM measures the particle number concentrations for particles between 0.25 and 32 µm in diameter in 31 classes and the dust mass concentration of the fractions PM_{10}, $PM_{2.5}$ and $PM_{1.0}$. Air and dust are aspirated in a rate of 1.2 L min^{-1} and analyzed in an optical cell. Then the air is sucked through a PTFE filter with a diameter of 47 mm and a pore size of 1.5 µm, on which all particles are collected. After each wind erosion event the filters were changed. Since the air stream does not hit the filter concentrically, but close to the edge, this results in an uneven covering of the filter with particles. On the one hand, this affects the density of cover, but on the other hand, sorting also already takes place, since the coarser fractions settle close to the inlet and the finer fractions are distributed over the entire surface of the filter. Therefore we always choose two sections from each filter for our analyses, one from the immediate vicinity of the inflow and one that is opposite from the inlet with the largest distance. Our results presented in this study are based on six erosion events that occurred between August to December 2016 (Table 1). For the EDM, a relative humidity of <60% is specified as an operation limit. Otherwise, moisture-induced aggregation of the individual particles may occur. All measurements were below this value; on 12/12/2022 the air humidity was even <20%.

These erosion events were already the basis of other investigations. Details regarding the spatial variability of the

TABLE 1 Description of the erosion events of this study.

Date	Start-end of event hh:mm	u > 6 m s^{-1} (hh:mm)	Wind direction	Mean wind velocity (m s^{-1})	Wind force integral[a]	EF[b]	DAS[c]	Soil loss kg ha^{-1}
08/26/2016	09:52–15:10	04:47	SSW	8.0	23,049	n.d	n.d	244
09/13/2016	09:25–15:39	06:14	SSW	9.6	119,559	52	74	408
11/18/2016	09:20–14:40	04:51	N	8.1	39,510	60	71	-11
11/21/2016	10:50–17:40	06:03	SSE	7.1	34,154	54	81	3
12/04/2016	10:16–19:10	06:41	NNE	8.5	75,120	61	67	24
12/12/2016	14:52–19:10	04:14	WSW	8.6	56,630	60	69	443

[a]based on the equation to calculate the transport capacity of the wind with WFI = ∑((u-ut)u2), for ut > 6 m s-1.
[b]erodible fraction estimated by sieving through 0.84 mm mesh.
[c]dry aggregate stability calculated following Skidmore et al. (1994).

TABLE 2 Results of the visual analyses of filter loadings by particle counting.

Date	Total counted particles	Number of phytoliths	Number of particles with biological origin	Per cent of phytoliths	Per cent of particles with biological origin
08/26/2016 IN	511	25	69	4.9	13.5
08/26/2016 OUT	523	31	87	5.9	16.6
09/13/2016 IN	1,210	53	183	4.4	15.1
09/13/2016 OUT	1,171	132	133	11.3	11.4
11/18/2016 IN	1860	155	452	8.3	24.3
11/18/2016 OUT	673	56	136	8.3	20.2
11/21/2016 IN	709	10	7	1.4	1.0
11/21/2016 OUT	1,414	47	3	3.3	0.2
12/04/2016 IN	211	16	0	7.6	0.0
12/04/2016 OUT	550	13	1	2.4	0.2
12/12/2016 IN	493	23	0	4.7	0.0
12/12/2016 OUT	778	170	0	21.9	0.0

transport processes at the field, the horizontal and vertical fluxes of the dust fractions and the influence of wind fluctuations on the dust concentrations can be found in Siegmund et al., 2018, Siegmund et al., 2022a and Siegmund et al., 2022b.

Scanning Electron Microscopy und Energy Dispersive X-ray analysis

Scanning electron microscopy (SEM, ZEISS EVO MA10) and Energy Dispersive X-Ray analysis (EDX, Bruker XFlash Nano 5010) were used to investigate the sampled dust on the filters regarding its size distributions in forms of singular particles or as part of aggregations. The filters of the EDM from 1 to 4 m height, at the windward and leeward site and of each event were analyzed separately by cutting out parts of the filters or by picking up the dust from the filters with adhesive stubs. The coarse particles had to picked up mainly with the stubs, because they could become released in the SEM due to the electrical charge. All samples were coated with Au/Pd to make the sample surface conductive for better resolution.

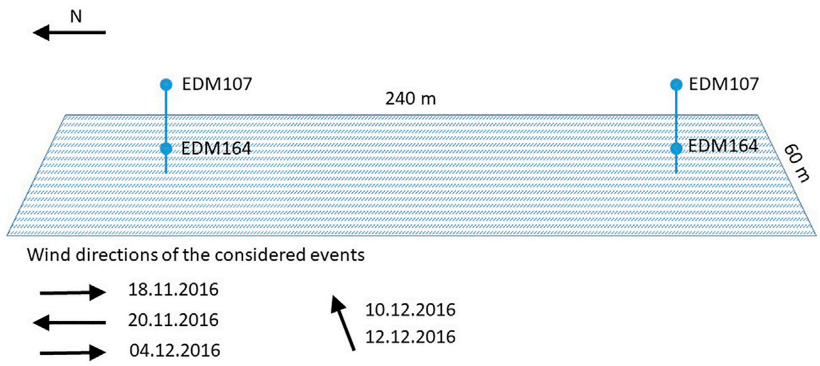

FIGURE 1
Setup of the dust measurements at the field site, arrows mark the wind directions of the regarded erosion events.

The classification was done visually by checking size and composition of the trapped particles. Image segments were selected that contained approximately 500 particles. After we could determine that a considerable part of the particles consisted of phytoliths, we compared identified phytoliths in the SEM images with similar already described morphotypes. For this purpose, the International Code for Phytolith Nomenclature (ICPN) 2.0 was used, which classifies opal phytoliths into 19 morphotypes defined by shape, size and surface ornamentation (Neumann et al., 2019). The particles identified as phytoliths were counted and set in proportion to the total number of particles of the image section. A distinction between the individual morphotypes was not made. Subsequently, single particles and image sections were analyzed by EDX, where matches with the visual analysis were checked.

Results and discussion

Generally the dust sampled at all days with wind erosion events shows great differences in the particle sizes and the particle composition. The EDM collected particles in a very wide size range, from diameters >100 µm to the sub-micron range of <1 µm in rounded, angular or elongated shapes. The number of particles increase exponentially with decreasing diameters. Particles of the PM_{10} -fraction were mostly below numbers of 10, while the EDM counted tens of thousands of particles of the 0.25 µm -fraction, which ultimately contribute little to the total mass. Thus, the comparative visual assessment can only be made within a size class by comparing the proportions of particles of approximately the same size. This is advantageously supported by the sorting processes during wind erosion. The particle size distributions of the filter loadings clearly reflect the different measurement heights. The larger particles with diameters around 100 µm, which are even outside the measuring range of the EDM of 0.25–32 µm, were predominantly transported in 1 m height (Figure 2, left). They are clear components of the saltation and short term suspension transport modes. At 4 m we could hardly find them on the filters and the particle sizes are smaller by one order of magnitude and more homogeneous (Figure 2, right). The differences in the concentrations of the different PM fractions are also reflected on the filter loading.

A closer look at the particles on the filters showed that a large part of them were of biological origin or possibly phytoliths. This was not surprisingly, since our measuring field was surrounded by other agricultural used areas, including pasture, reduced and conventional tilled fields, and the soils in La Pampa have developed under forest and grass steppes. Thus, the separate consideration of our pictures under this aspect was obvious. Table 2 summarizes the results of the visual analyses counting particles on the filters of the six erosion events.

It is noticeable that in the first three events the proportion of organic components is considerably higher than in the last three events. The proportion of phytoliths also tends to be lower. This can be explained by the fact that these three erosion events were always the first erosion events immediately following a tillage operation. These were carried out to remove vegetation at the plot and create a susceptible surface by breaking crusts. The last tillage occurred on 11/17/2016, and due to the persistently dry weather, no further tillage was necessary. The following erosion events consequently occurred on an already depleted soil surface, where especially the most easily erodible components were removed. These effect was also shown by Münch et al. (2022), who could prove that especially fresh applied or only slightly decomposed organic matter will be blown out by the wind at first.

As wind erosion is also a sorting process, its occurrence results in different particle size distributions of the eroded material in comparison to the original soil. Our studies show for the three erosion events with the initial field conditions,

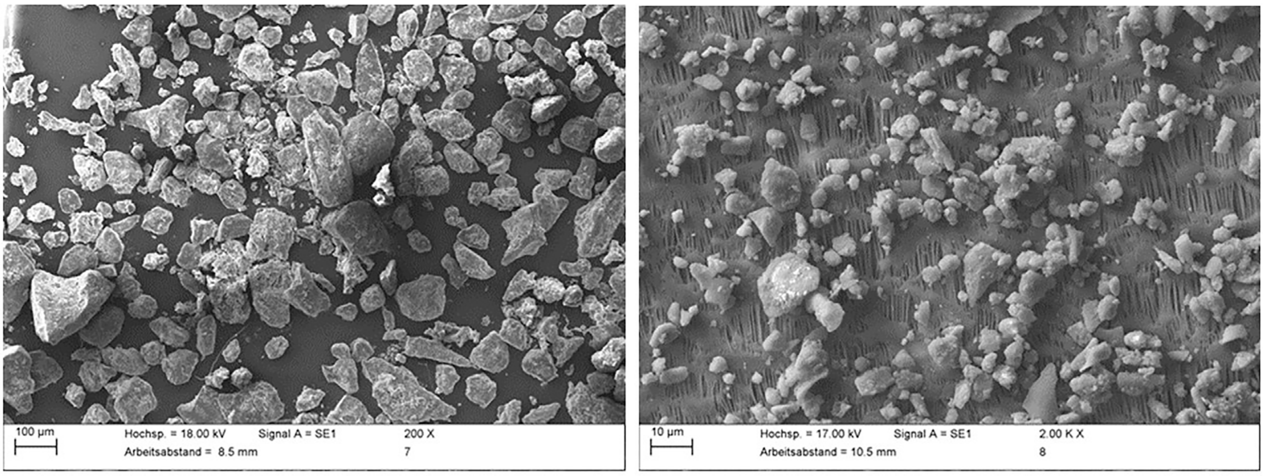

FIGURE 2
Overview of particles sampled on filters of the EDM in 1 m height (left, on an adhesive stub) and 4 m height (right, on a cut-out of the filter), scale bars in the lower left corner.

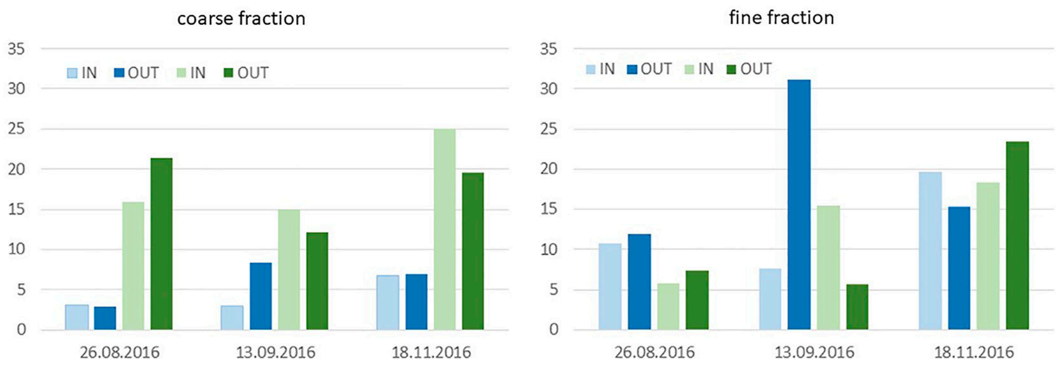

FIGURE 3
Percentage of phytoliths (blue bars) and particles of organic origin (green bars) in the coarse and fine fractions of the dust.

that there were differences in the composition between the coarse and fine fractions of the particulate matter. In the coarser fraction, particles of organic origin, as plant residues or residues in its early stage of decomposition, were more prevalent, while phytoliths were significantly less represented. In the finer fractions, the phytoliths were in the majority or at least at the same level (Figure 3, Figure 4). The proportion of phytoliths corresponds to those of other authors and ranges from 3 to 8.5% in the coarse particle fractions and from 10 to 31% in the fine particle fractions. This leads to a relative enrichment of the organic matter in the coarse dust fraction, since the sorting takes place only by aerodynamics of the very diverse shaped or composed particles. Thus, the enrichment of organic matter in the dust can be derived predominantly from the shift in size composition between organic and mineral particles. Phytoliths are much more similar to mineral particles in shape and density and are therefore subject to the same sorting and transport mechanisms.

Latorre et al. (2012) already pointed out that the majority of the windblown phytoliths in dust from the Pampean plains could be derived from grass species or other plant communities grown in the region, and that the highest concentrations could be found during periods of increased agricultural activity and bare soils. Thus, there should be a relationship between the releasing processes, such as wind erosion or tillage, and the amount of phytoliths. Since we had erosion events ranging from weak to medium intensity on our measurement field, we

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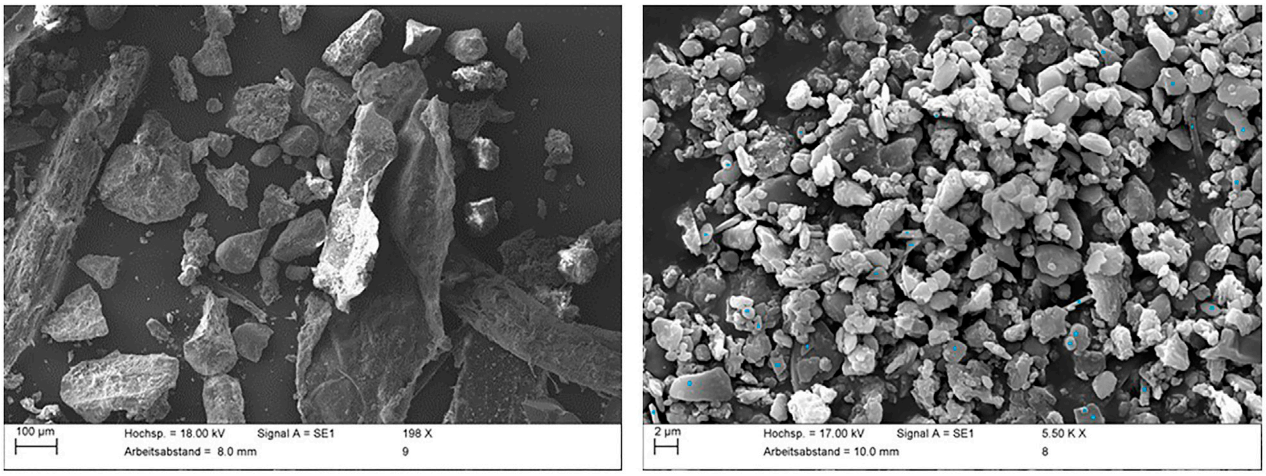

FIGURE 4
SEM images of the coarse and fine fractions of the sampled dust; left image shows big particles of organic origin, in the right image are phytoliths marked with blue dots.

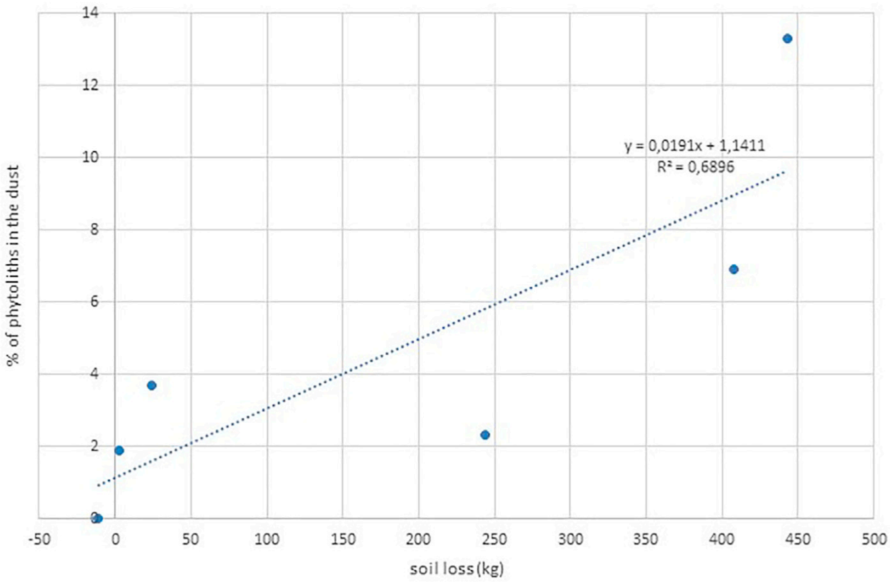

FIGURE 5
Percentage of phytoliths in the dust in dependence on wind erosion intensity.

combined the data from the wind erosion measurements with the phytolith estimation from the filters of each event. The relationship between the measured soil losses of all wind erosion events (Table 1) and the percentage of phytoliths in the dust shows a tendency of increasing shares of phytoliths with increasing soil losses (Figure 5). Thus, firstly an increasing ratio between emitted particles and the proportion of phytoliths can be derived and secondly it shows again that phytoliths are effected by the same sorting processes as mineral particles, because they are predominant in the finer dust fractions together with mineral particles of the same size. So, agriculturally used areas can be an important source for airborne phytoliths in the PM fractions which are released by wind erosion.

Since visual determination of the phytoliths in the total filter loading is despite the inclusion of determination keys subject to subjectivity, we used EDX to try to eliminate this as much as possible, to identify more precisely the particles we refer to as phytoliths. Since only visual clues could be used for the phytolith classification, EDX can be used to validate these analyses. Since minerals also contain silicon, other elements must help to distinguish them from phytoliths. These can be aluminum, iron or sodium, which can be clearly assigned to mineral particles. Thus, in this case only one particle at the lower right edge of this image can be identified as phytolite origin by this exclusion method. Figure 6 shows an example of a larger section of a filter, where we counted about 140 particles and classified about 10% as phytoliths. Element mapping and applying the same exclusion methodology shows a quite good agreement between the classification and the verification. Thus, the purely visual assignment of phytoliths *via* the morphotypes can be considered sufficiently accurate. Problems arise mainly with the very small particles. Here, unfortunately, the EDX does not provide the same resolution as the SEM.

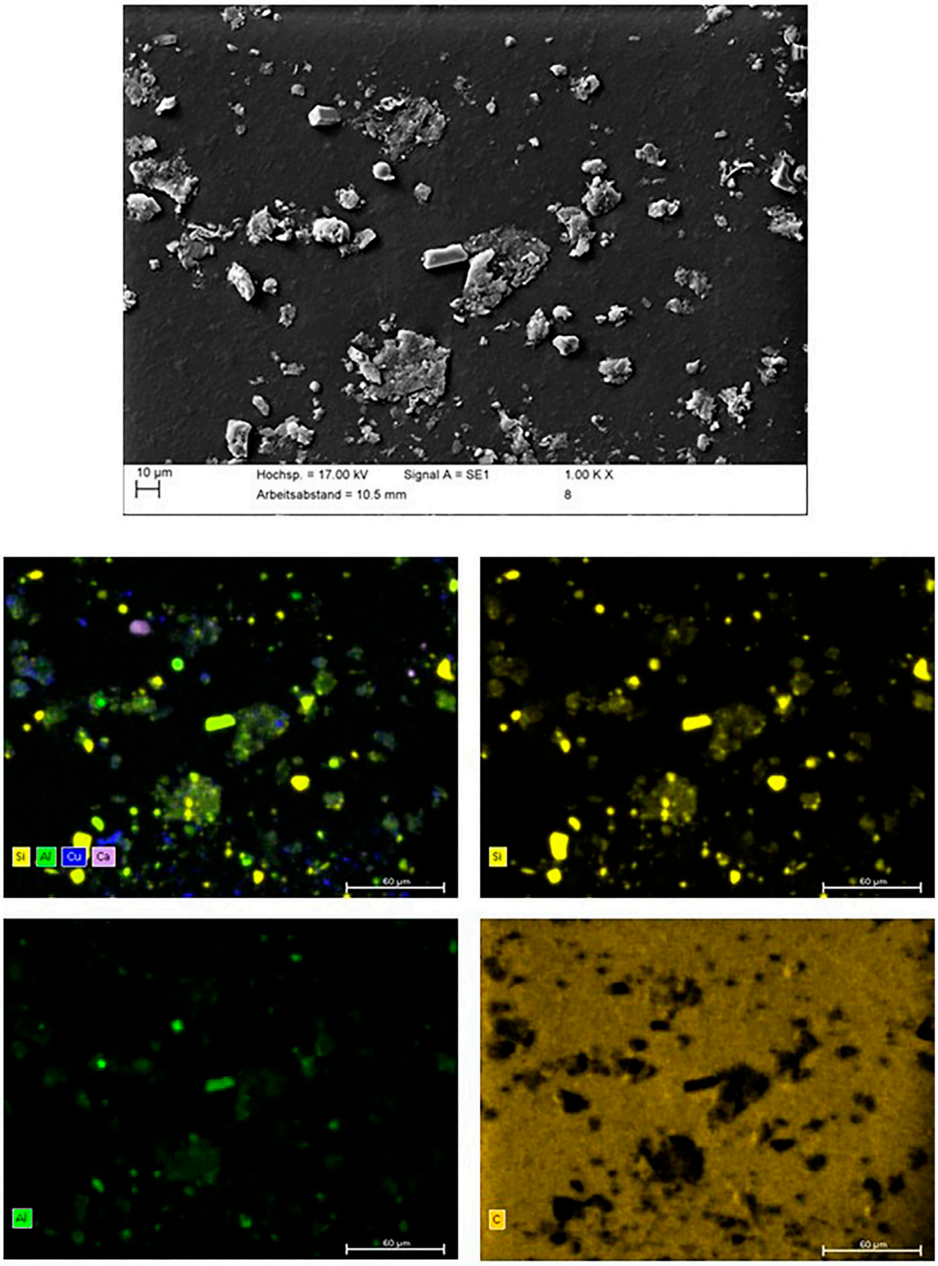

FIGURE 6
SEM and EDX analyses of a dust sample containing phytoliths.

Conclusion

SEM analyses provide extremely detailed insights into composition and shape diversity of dust samples. The complex composition of dust particles sampled during wind erosion events makes it difficult to assign them certain properties. Although a large number of studies have demonstrated an enrichment of organic matter as well as essential elements in suspended PM, there is still some uncertainty because a complete separation does not occur. The enrichment of organic matter can be clearly attributed to the shift in particle size composition after aeolian sorting if mineral and organic particles are defined by their aerodynamic diameter. Especially these larger particles of biological origin are also the carriers of mineral adhesions in the micro- and sub-micro scale. In the reverse case, mineral particles are known to carry "hitchhiking" microorganisms (Giongo et al., 2012; Favet et al., 2013). Thus, the emitted dust always reflects the diversity of properties of its origin and the special properties of agricultural dust, such as the higher ice nucleation activity, cannot be attributed to separated particles.

In our investigations, we were also able to detect phytoliths in the dust as well as their accumulation in the PM fractions. Since we could not separate the phytoliths by appropriate pretreatments, we had to identify them in the bulk sample. A check by means of elemental analysis with EDX showed only small deviations from our assignments, so we can conclude that most of the phytoliths were recognized by their morphotypes. Most recently, research has focused on silicon as being important for soil water holding capacity and water availability in soils (Schaller et al., 2020). The removal and transport by aeolian processes could be proven with our investigations. Therefore, particularly in the semi-arid arable areas, increased attention should be paid to any possibility of improving the water availability of the soils used there.

Data availability statement

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

Author contributions

Conceptualization, methodology, RF and MS; funding acquisition, RF, MS, and DB; investigation, NS, JP, FA, and LI; REM and EDX analyses, image processing JB and RF; writing—original draft RF; review and editing, all co-authors. All authors have read and agreed to the published version of the manuscript.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Publisher's note

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