

Assessing the oxidative potential of dust from great salt Lake

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HIGHLIGHTS

- The GSL has reached historically low levels.
- As and Li levels in GSL exceeded EPA residential regional screening levels.
- The oxidative potential (OP) of dust from GSL is higher than the OP in other regions.
- The OP of the GSL dust was associated with metals, including Cu, Mn, Fe, and Al.
- The choice of OP assay and extraction solvent affects the OP activity of redox-active species.

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ABSTRACT

Great Salt Lake (GSL) is one of the world's largest inland bodies of saltwater. Climate change, drought, and water diversions have led to the GSL reaching historically low levels, exposing over 1945 km² of lakebed. Dust events can transport particulate matter (PM) from the GSL's exposed lakebed to the adjacent urban areas, presenting air quality and health concerns. In this study, we assessed the oxidative potential (OP) of dust from the exposed GSL bed compared to dust samples collected from other regional playas and to reference Arizona test dust. The oxidative potential of PM₁₀ (particles ≤10 μm in aerodynamic diameter) derived from these dusts was assessed by two acellular assays: the ascorbic acid (OP^{AA}) and dithiothreitol (OP^{DTT}) and two extraction solvents: phosphate buffer saline and Gamble's simulated lung fluid. All samples were also analyzed for elemental composition. Our results show that the concentration of As and Li in most of the GSL dust samples exceeded the Environmental Protection Agency (EPA) soil residential regional screening levels, and the OP^{AA} and OP^{DTT} of dust samples from GSL playas were generally higher than the OP of other regional playas. Our findings also indicated that the OP of the GSL dust was associated with metals, including Cu, Mn, Fe, and Al. This is the first study evaluating the oxidative potential of dust from the GSL, which improves our understanding of how increasing aridity, particularly desiccation of saline lakes, affects air quality and public health.

1. Introduction

The global increase in arid and semi-arid lands due to climate change (Huang et al., 2016; Song et al., 2018) has led to increased ambient dust levels and dust events (Achakulwisut et al., 2018; Schweitzer et al., 2018). Dust events negatively influence air quality and human health (Chen et al., 2021; De Longueville et al., 2010); such dust events occur when wind speeds surpass the threshold required for particles to be lifted from the soil (Jayaratne et al., 2011; Lei and Wang, 2014). Dust storms can transport harmful metals and metalloids, such as arsenic and lead, over long distances from the source region (Goudie, 2014; Morman

and Plumlee, 2013). Previous studies have associated human exposure to dust events with adverse health effects, including lung cancer and cardiovascular mortality (Anderson et al., 2012; de Longueville et al., 2013; Zhang et al., 2016).

Increasing dust storms are a concern in the Salt Lake Valley due to the shrinking of the Great Salt Lake (GSL). GSL, which lies to the west of Utah's densely populated Wasatch Front (2.6 million people) (Nehra and Caplan, 2022), reached a historic low-level of 1275 m in 2022, with an exposed lakebed of over 1945 km² (Lang et al., 2023; Wurtsbaugh and Sima, 2022). Dust from the GSL contains enhanced levels of heavy metals, such as, Sb, and Cu, at levels that exceed the Environmental

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Protection Agency (EPA) regional screening levels (Perry et al., 2019; Putman et al., 2022). On average, four to five dust storms occur in northern Utah annually (Hahnenberger and Nicoll, 2012; James Steenburgh et al., 2012); such dust events transport metal-contaminated dust from the GSL and present a concern for the air quality and health in the adjacent urban areas. Despite these concerns, no study has examined the impacts of dust from the GSL on human health.

Epidemiological studies in Utah's Wasatch Front have established connections between exposure to particulate matter (PM) and the heightened occurrence of hospital admissions due to respiratory and cardiovascular diseases (Brook et al., 2010; Horne et al., 2018; Leiser et al., 2019; Pope et al., 2008). Toxicological studies have also linked airborne dust exposure to adverse biological effects (Atafar et al., 2019; Courter et al., 2007; Faraji et al., 2018). However, the mechanisms for dust-related health and biological effects are not completely understood, although several studies have suggested that these effects could be related to dust's oxidative potential (Chirizzi et al., 2017; Lovett et al., 2018; Pant et al., 2015).

Oxidative potential (OP) measures the capacity of PM to generate reactive oxygen species (ROS) (Borlaza et al., 2018a; Yadav and Phuleria, 2020). The OP of PM has been reported as a more relevant metric to screen for biological effects than PM mass because several biologically significant properties such as size, composition, surface area, and bioaccessibility contribute to the OP of PM (Gao et al., 2020b; Janssen et al., 2014; Yang et al., 2015). Studies have associated ROS formation as a possible pathway linking dust exposure and adverse health effects, such as lung cancer and asthma (Li et al., 2003; Rogers and Cismowski, 2018). ROS are radicals or molecules with a single unpaired electron in their outermost shell of electrons (Bayir, 2005; Halliwell, 1992); the presence of unpaired electrons results in the high reactivity of ROS. Oxidative stress occurs when the generation of ROS exceeds the available human antioxidant defenses, which can lead to inflammation and damage to lipids, proteins, and DNA structures (Khansari et al., 2009; Schieber and Chandel, 2014; Sosa et al., 2013). Previous studies on PM-associated adverse health effects in Utah were focused on anthropogenic PM sources like transportation and industrial emissions (Ghio, 2004; Ou et al., 2020; Yu et al., 2023). Information regarding the OP of natural PM sources remains limited, and there is currently no available data regarding the OP of dust from the GSL. This study aims to fill this gap by evaluating the OP of GSL dust and comparing these to dust from regional playas.

The two widely used acellular assays to measure OP are the dithiothreitol (DTT) assay (Cho et al., 2005) and the ascorbic acid (AA) assay (Mudway et al., 2005) because they are cost-effective, have high reproducibility, and are a good indicator of adverse biological effects (Bates et al., 2019; Jiang et al., 2019; Ma et al., 2021). Assessing OP by DTT (OP^{DTT}) or AA (OP^{AA}) is based on the ability of redox-active components in PM to oxidize DTT (a surrogate for cellular antioxidants) or ascorbic acid (the most abundant antioxidant in lung fluids), respectively, by catalyzing the transfer of electrons from DTT or AA to molecular oxygen thereby promoting the formation of ROS (Fang et al., 2017a; Gao et al., 2020a).

Redox-active metals in dust particles have been linked to ROS formation and activating antioxidant-signaling pathways (Pardo et al., 2017; Rodríguez-Cotto et al., 2015). Water-soluble transition metals, such as Fe and Cu, participate in redox-cycling reactions, which may enhance DNA damage (Stohs, 1995; Valavanidis et al., 2005; Valko et al., 2005). Several studies have reported correlations between metals in urban dust (e.g., Ni, Cu, Cr, Mn, Cu, and As) with high OP^{DTT} and OP^{AA} (Chirizzi et al., 2017; Kajino et al., 2021; Nishita-Hara et al., 2019; Pant et al., 2015). Nishita-Hara et al. (2019) observed an increase in the particle-bound OP^{DTT} in PM_{10} (aerodynamic diameter less than 10 μm) dust samples from western Japan strongly correlated with the mineral dust-derived elements (Cu, Al, Fe, Mn, Mg), suggesting that the concentration of mineral dust elements is an important controlling factor for the OP of coarse particles. Borlaza et al. (2018) revealed that OP

activities of PM_{10} sampled from Korean sites increased during the spring and summer, possibly due to increased metals (Al, Fe, Mn, V, and Zn) caused by dust events. Mallone et al. (2011) also reported epidemiological evidence linking PM_{10} to elevated mortality rates during dust storm events in Italy; they attributed these findings to heightened exposure to metals in dust transported from the Sahara.

This study aims to (1) investigate the OP of 10 $p.m._{10}$ dust samples (5 GSL, 4 from other regional playas, and reference Arizona test dust) using the DTT and AA assays and (2) compare the two OP methods (OP^{DTT} and OP^{AA}) to evaluate the sensitivity of the two OP methods to individual elements in the dust samples, (3) characterize the composition and bioaccessibility of the dust, and (4) correlate the OP and composition to identify components and sources contributing to the OP.

2. Methodology

2.1. Dust sample collection and preparation

Dust samples were collected from the exposed lakebed of the GSL and other regional playas (Sevier Dry Lake (SD), Tule Dry Lake dust (TD), Fish Springs Lake (FD), and West Desert (WD)) as shown in Fig. 1. Perry et al. (2019) performed a comprehensive examination of the GSL's exposed lakebed using an incremental sampling methodology (ISM). In accordance with ISM guidelines, they subdivided the lakebed into 10 Decision Units (DUs), each consisting of 122 subunits (SUs), as shown in Fig. 2. The longitude and latitude of the sample locations are also shown in Fig. 1. These samples were dried in an oven at a temperature of 105 °C for 24 h, followed by disaggregation and sieving to obtain dust samples with particle sizes less than 74 μm . Details on the site sampling procedure, preparation, and separation are provided in Perry et al. (2019). Arizona Test Dust (AD), a commonly used surrogate of atmospheric mineral dust for toxicology studies (Cote et al., 2018; Ichinose et al., 2008; Nishita-Hara et al., 2023), was used as reference dust.

It is essential to evaluate OP and bioaccessibility with inhalable PM, specifically PM_{10} ; therefore, a system was developed to further separate PM_{10} samples from the coarse dust samples (<74 μm), as shown in Fig. 3. The coarse dust samples were placed into a flask and aerosolized using compressed air. The flask was placed on a magnetic stirrer to enhance the fluidization of the particles. The coarse dust samples passed through a MiniVol™ Tactical Air Sampler (Airmetrics USA), which contained a PM_{10} inlet separator and impactor to separate PM_{10} size fractions. The PM_{10} dust sample was collected on a 0.5 μm , 47 mm PTFE filter (Whatman, USA) at a flow rate of 5 LPM. This study characterized the composition and OP of the dust samples, and for the GSL, we selected five GSL dust samples (DU2-SU7, DU5-SU3, DU5-SU-4, DU5-SU10, DU9-SU14) to represent the range of the GSL samples. In total, we used 10 dust samples (5 GSL, 4 regional playas, and reference AD) for compositional and OP analysis.

2.2. Elemental composition analysis

Dust samples were treated using four different leaching solutions: phosphate buffer solution (PBS), Gamble's solution synthetic lung fluid (SLF), 1 M ammonium acetate buffer at pH = 7.0, and 1 M acetic acid. PBS was obtained from Sigma-Aldrich and treated with Chelex. SLF was obtained from Biochemazone and kept frozen until use; Ammonium acetate buffer and acetic acid solutions were prepared from concentrated ammonium and glacial acetic acid, both trace metal grade. The leaching treatment was done in parallel; each dust sample was split into four sub-samples, mixed with each leaching solution, and analyzed in the following way. Approximately 10 mg of dust for each sample/leaching solution was weighed in a 1.5 mL acid-leached polypropylene centrifuge tube, and 0.5 mL of each leaching solution was added and weighed. The vials were vortexed and then kept on a shaking table for 24 h at room temperature. The vials were then vortexed again and centrifuged at 10,000 rpm for 5 min. The clear supernatant leachates

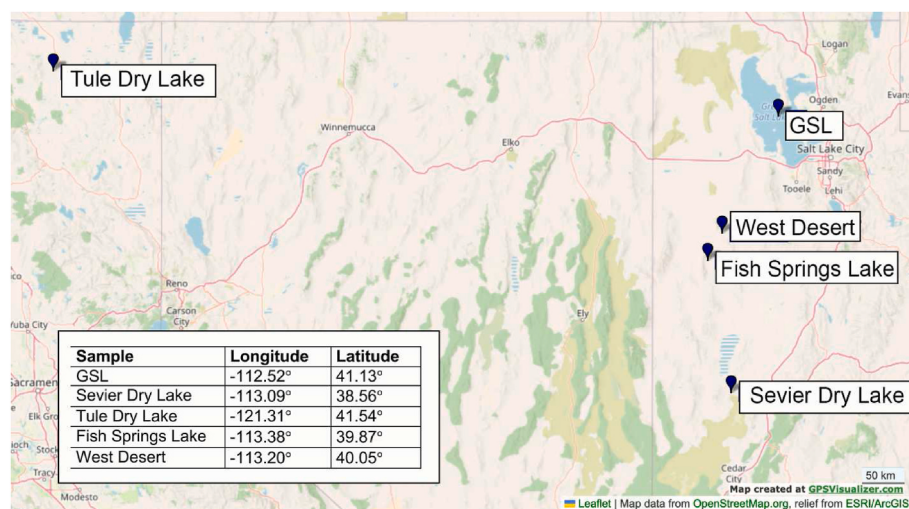


Fig. 1. Map showing the location of sample sources.

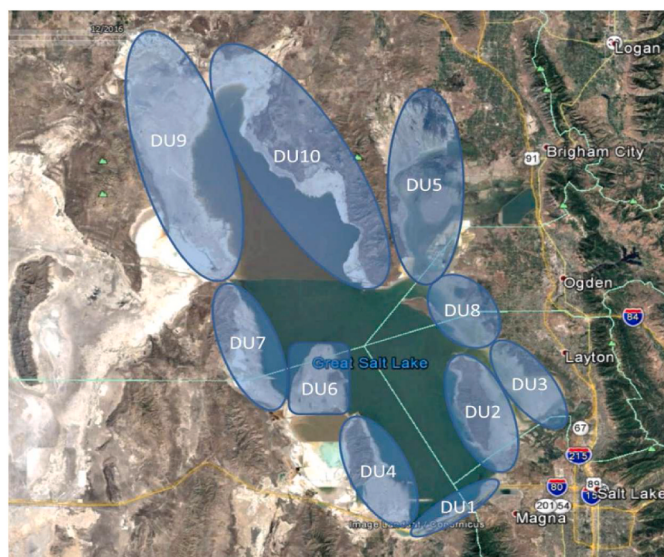


Fig. 2. Map showing the decision units (DUs) along the Great Salt Lake (Perry et al. (2019)).

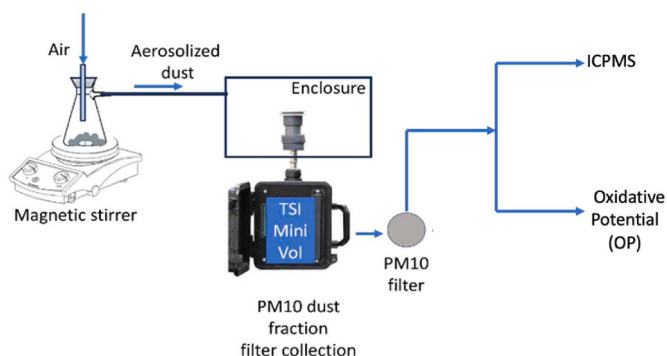


Fig. 3. Experimental setup to separate PM₁₀ from the PM₇₄ size fractionated dust samples.

were then diluted as follows: an aliquot of 0.100 mL of leachate was transferred into a polystyrene 14 mL tube, then acidified with 0.250 mL of concentrated HNO₃ (trace metal grade) and diluted with Type I water

(IQ 7000, MilliporeSigma, Burlington, MA, US) to 10.0 mL. In this way, the total dilution factor was 5000. Elemental quantification was performed with a triple quadrupole inductively coupled plasma mass spectrometer (ICP-MS, Agilent 8900, Santa Clara, California). An external calibration curve was prepared from 1000 mg/L single element standards (Inorganic Ventures, Christiansburg, VA. Elements determined were Li, Be, Na, Mg, Al, K, Ca, Sc, Cr, Mn, Co, Ni, Cu, Ti, Zn, V, Fe, Ga, Rb, Sr, Y, As, Mo, Ag, Cd, Sb, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb, Lu, Tl, Pb, Bi, W, Th and U. Samples, calibration solutions, and blanks were added 10 ng/mL indium as internal standard and run in the ICP-MS using a dual-pass quartz spray chamber, PTFE nebulizer, dual-syringe introduction system (Teledyne, AVX 71000), platinum cones and sapphire injector in a platinum-shielded quartz torch. The limits of determination (LoD) were calculated as three times the standard deviation of the blanks, multiplied by the total dilution factor used for each sample. The instrument is in a filtered air positive pressure lab, and sample handling and dilutions were performed in laminar flow benches using calibrated pipettors (Eppendorf Reference, Hamburg, Germany). Data quality for both ICPMS quantification was determined by using Certified reference materials 1643f (Trace elements in water, National Institute of Standards and Technology, Gaithersburg, MD, with measured values within 5% of the certified values.

2.3. Oxidative potential analysis

The OP of the PM₁₀ dust samples was assessed by both DTT and AA acellular assays. We also evaluated the effect of the extraction solvent on the OP of the dust samples by extracting the dust samples in two different aqueous solvents: phosphate buffered saline (PBS, pH = 7.4) and Gamble's solution (Biochemazone, pH = 7.4), which is a simulated lung fluid (SLF) solution used for accessing the bioaccessibility of PM in physiological fluids (Calas et al., 2017; Julien et al., 2011; Pietrogrande et al., 2021). The PBS was treated with Chelex® 100 resin (sodium form, BioRad, USA) to remove any trace metal ions that may catalyze oxidative reactions and to reduce background oxidation of DTT and AA.

Sample preparation: The PM₁₀ particles were carefully scraped from the filter, weighed, and suspended in a glass vial using the two solvents. The suspensions were sonicated in an ultrasonic bath sonicator (Branson 2800, USA) for 15 min. A total of 1 mg/mL of the suspended dust PM₁₀ sample solution was diluted to 100 µg/mL in PBS and SLF. The dust suspension was not filtered to include both soluble and insoluble species that may contribute to the total OP of the dust particles. Such a method has been used to study the OP of both soluble and insoluble species components of PM in physiological conditions (Borlaza et al.,

2021; Daellenbach et al., 2020; Fang et al., 2017b; Gao et al., 2017).

Reagent preparation: Solutions of DTT and 5,5'-dithiobis-(2-nitrobenzoic acid) (DTNB) (Sigma-Aldrich, USA) were prepared in PBS at a concentration of 0.75 mM and 1 mM, respectively. Solution of AA (A92902 Sigma Aldrich, USA) was also prepared in PBS at the same concentration as DTT (i.e., 0.75 mM). The reagent solutions are sensitive to light. As a result, they were stored in vials wrapped in aluminum foil and kept in a dark environment throughout the experiment. A 35 µg/mL H₂O₂ and just PBS were used as positive and negative controls (referred to as blank), respectively.

DTT assay: 100 µL of the 0.75 mM DTT solution was added to 500 µL of 100 µg/mL of the dust samples (suspended in PBS (OP^{DTT} PBS) and SLF (OP^{DTT} SLF)), negative and positive control in different 1.5 mL vials at defined time points (0, 10, 20, 30, and 40 min). The mixture was vortexed and placed in an incubator (VWR, USA) under controlled conditions of 37 °C and pH of 7.4. At the end of the final time point of 40 min, the reaction vials were spun at 1.2k xg for 3 min using a micro-centrifuge (AccuSpin Micro 17R, Fisher Scientific USA) to remove particles that might interfere with the absorbance reading. A 100 µL aliquot of the spun solution was transferred to a 96-well plate (Greiner Bio-One UV-Star Polystyrene) in triplicate, after which 50 µL of 1 mM DTNB solution was added to each aliquot. The negative control (blank) and positive control were run in duplicates. The unconsumed DTT reacts with DTNB to form a colored product, 2-nitro 5-thiobenzoic acid (TNB); the absorbance of the TNB was measured at a wavelength of 412 nm, using a plate reader (Molecular Devices, CA, USA).

AA assay: 25 µL of AA solution was added to 100 µg/mL of the dust samples (suspended in PBS (OP^{AA} PBS) and SLF (OP^{AA} SLF)) in a 96-well plate (Greiner Bio-One UV-Star COC). The rate of AA consumption was directly measured in the plate reader at 265 nm at a time interval of 10 min for 2 h. The 96-well plate was auto-shaken for 60 s before each absorbance reading, and all the sample absorbance measurements were done in triplicate. The absorbance from the dust samples without any AA was also measured and subtracted from the final sample absorbance readings to remove the contribution of the background particle absorbance.

The sample's DTT or AA consumption rate (n_s , pmol/min) was determined from the linear slope and intercept of measured absorbance versus time. We limited DTT and AA consumption to less than 20% of the initial concentration to achieve a linear consumption rate. The consumption rate (n_s) is given by:

$$n_s = -\sigma_{abs} \times \frac{N_0}{Abs_0}$$

Where σ_{abs} is the slope of absorbance versus time, N_0 is the initial amount of DTT or AA in pmol, and Abs_0 is the initial absorbance calculated from the intercept of linear regression of absorbance versus time.

OP of the sample (pmol/min/µg) is defined by the DTT or AA consumption rate normalized by sample mass added to the reaction mixture. This is given by:

$$OP_{sample} = \frac{n_s - n_{blank}}{m_{sample}}$$

Where n_{blank} is the rate of DTT or AA consumption in the blank, and m_{sample} is the mass of the sample in the reaction mixture.

2.4. Data analysis

We used a one-way analysis of variance (ANOVA, [astatsa.com](https://www.astatsa.com) online) to assess differences in OP among the ten samples. Subsequently, we conducted a post-hoc Tukey HSD test to identify OP differences between dust sample pairs (PBS and SLF). The results are displayed as the standard deviation of the mean. Significance levels of $p < 0.01$ and $p < 0.05$ were used to determine statistical significance. We also conducted a

Pearson correlation analysis to assess the relationships between OP responses in various OP assays (OP^{DTT} and OP^{AA}) and the correlation between different extraction solvents (PBS and SLF). Additionally, we used principal component analysis to investigate the association between OP responses and metal concentrations in the dust samples.

3. Results and discussion

3.1. Elemental concentrations of dust samples

Table 1 shows the elemental concentrations in SLF-extracted dust samples. The SLF-extracted elemental concentration enables investigation of the bioaccessibility and fate of deposited dust particles in lung fluid. Table S1 (Supplementary material) contains a more comprehensive list of the mass composition of the dust samples as measured by ICPMS. The United States EPA developed regional screening levels (RSLs) as a reference for evaluating the ecological risk associated with soil contaminants. The first step in our analysis involved comparing our dust composition measurements against these RSLs. We removed all elements (Be, Co, Ni, Se, Cd, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Yb, Lu, Th) that had mass concentration values below their minimum detection limit (MDL). Out of the 24 remaining elements, 8 were not found in the EPA RSL database, suggesting that there is insufficient information to assess health concerns linked to these elements. These 8 elements include Na, Mg, Ca, K, Rb, Y, Cs, and S.

The metal concentrations, shown in Table 1, indicate a high concentration of crustal elements (Na, Mg, K, Ca, and S). These crustal elements are associated with evaporated minerals that form on the surface of the lakebed (Alkhayer et al., 2023; Cabestrero et al., 2018; Levy et al., 1999). Various transition metals were detected in all the samples. The average concentration of transition metals (B, Mn, Fe, Cu, V) and metalloids (Li, Rb, As, Sb, Cs, Pb) from the GSL sites were higher than those in other regional playas. Arsenic is of particular focus at the GSL due to reports of its enhanced concentrations in dust aerosols and its known toxicity (Baxter and Butlerreditors., 2020; Castro-Severyn et al., 2020; Wurtsbaugh, 2012). Among the trace elements measured, lithium and arsenic concentrations exceeded the soil residential RSL in most of the GSL sites and other regional playas collected in this study, as shown in Fig. 4. Previous studies have also reported the enrichments of trace elements such as arsenic and lead at dust collection sites within the GSL and Wasatch front (Goodman et al., 2019; Putman et al., 2022; Reynolds et al., 2010; Thorsen et al., 2017).

3.2. OP using AA and DTT assays

Fig. 5 shows the OP^{AA} of all the dust samples from different sampling sites in PBS and SLF normalized by the sample mass. The OP^{AA}PBS and OP^{AA}SLF of GSL dust samples were generally higher compared to other regional dust playas, including the reference AD. Notably, the OP^{AA}PBS of the DU5-SU4 GSL dust sample was significantly higher than the OP^{AA}PBS of the AD and other regional playas ($p < 0.05$). The mean OP^{AA} PBS and OP^{AA} SLF of GSL samples were within the ranges of 3 ± 0.03 to 5.57 ± 0.14 pmol/min/µg and 1.95 ± 0.05 to 2.98 ± 0.14 pmol/min/µg respectively.

Fig. 6 shows higher OP^{DTT} of all the dust samples from different sampling sites in PBS and SLF normalized by the sample mass. The OP^{DTT}PBS and OP^{DTT}SLF of most of the GSL dust samples were also higher compared to other regional dust playas but lower than the OP^{DTT} of reference AD. Specifically, the OP^{DTT}PBS and OP^{DTT}SLF of the DU5-SU4 GSL sample were significantly higher than the OP^{DTT} of other regional playas for both solvents. Overall, the OP^{DTT} PBS and OP^{DTT} SLF of GSL samples were within the ranges of 1.75 ± 0.24 to 4.89 ± 0.5 pmol/min/µg and 1.15 ± 0.02 to 3.85 ± 0.07 pmol/min/µg, respectively.

Several studies have reported adverse biological outcomes from exposure to AD (Cote et al., 2018; Ichinose et al., 2008; Kameda et al.,

Table 1

Concentrations of selected elements (mg of element per kg of sample; mg/kg) in SLF leachate measured by ICPMS.

Sample	AD	WD	TD	SD	FD	DU2-SU7	DU5-SU3	DU5-SU4	DU5-SU10	DU9-SU14
Li	3.32	16.5	8.05	6.83	22	19.3	9.41	20.1	7.25	17.9
B	1	26.1	26.4	46.3	109	111	46.4	33.6	19.2	26.5
Na	22,001	32,806	27,308	33,149	34,454	41,974	47,375	35,468	44,188	38,838
Mg	282	1901	671	1610	2529	7386	2671	4859	1263	1315
Al	7.33	1	1	2.38	1.5	1	1.5	1.34	1.55	5.36
K	683	1924	3066	1934	2139	2928	3076	3685	1583	3132
Ca	717	1535	1556	3015	11162	3628	1649	1907	481	1956
Mn	1.61	0.66	0.28	0.15	0.60	1.12	0.42	1.75	0.80	1.69
Fe	25.4	39.6	6.97	8.59	14.2	9.26	11.68	84.2	3.32	19.8
Cu	0.05	0.14	<MDL	<MDL	<MDL	0.08	<MDL	0.45	<MDL	<MDL
V	1	0.52	0.84	0.43	0.28	0.22	0.28	0.55	0.63	1.15
Cr	0.43	0.80	0.13	0.18	0.23	0.14	0.17	1.36	0.05	0.29
Rb	0.89	2.88	4.35	1.17	9.07	7.76	9.27	7.73	2.91	6.50
Sr	27	155	138	84	340	151	81	46	63	77
Y	0.004	0.003	0.002	0.003	0.005	0.006	0.006	0.008	0.002	0.010
As	0.34	1.04	4.61	1.13	1.52	2.64	1.98	1.00	1.61	1.63
Mo	0.08	0.59	6.74	1.19	0.62	3.64	0.38	0.65	0.19	0.85
Sb	0.06	0.04	0.03	0.04	0.02	0.22	0.08	0.06	0.02	0.04
Cs	0.01	0.01	0.06	0.01	0.05	0.09	0.05	0.03	0.01	0.09
Ba	13.6	21.5	10.2	3.8	4.25	3.83	3.68	7.02	0.07	7.71
Tl	0.01	0.00	0.02	0.00	0.01	0.02	0.01	0.02	0.00	0.01
Pb	0.02	0.01	<MDL	<MDL	<MDL	0.05	0.03	0.03	0.00	0.04
U	0.12	1.84	2.28	1.37	2.31	1.90	1.21	0.69	0.81	0.67
S	132	6611	5056	9805	24,168	24,959	6961	7704	2086	2670

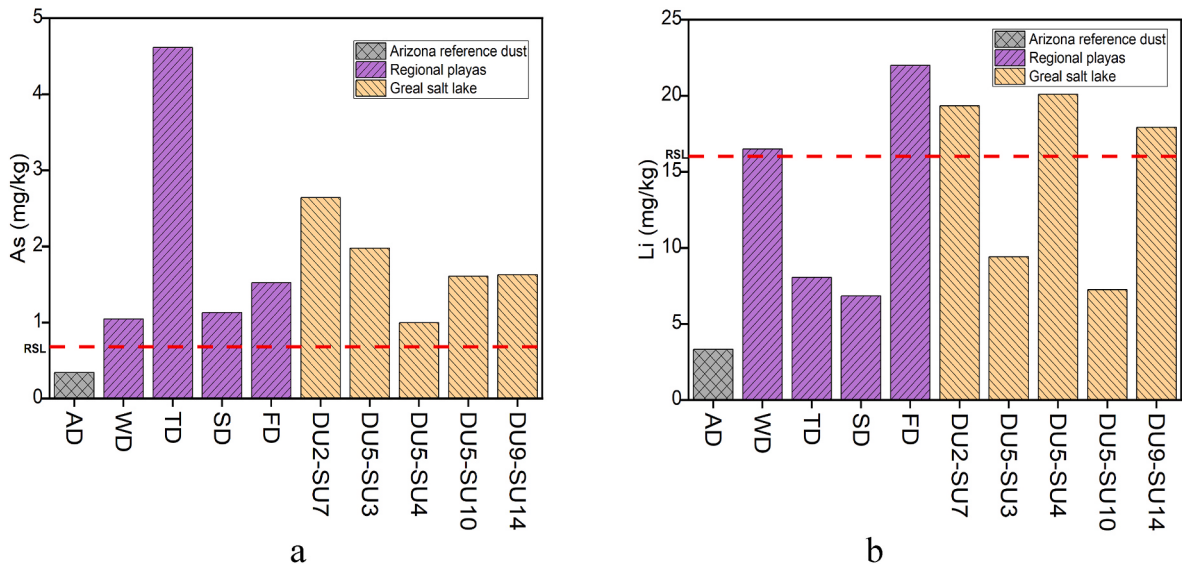


Fig. 4. Elemental mass concentrations (mg/kg) for (a) arsenic and (b) lithium compared with EPA-recommended soil residential screening levels determined by ICP-MS analysis in SLF extract. The red dashed horizontal line indicates the regional screening level for each element.

2016; Nishita-Hara et al., 2023). Ichinose et al. reported that AD caused a slight increase of neutrophils in bronchoalveolar lavage fluids and pro-inflammatory mediators, such as keratinocyte chemoattractant masters of the air. By comparing the OP of GSL and other regional playas to AD, we can assess the relative health risks associated with dust exposure from the GSL.

The OP^{AA} of some of the GSL samples, particularly DU5-SU4, were generally higher compared to AD samples and other regional playas. This suggests that the GSL contains more redox-active species than AD and other playas. In general, the measured OP^{DTT} of GSL samples in this study is within the range of previously reported OP values (3–10 pmol/min/ μ g) from dust sources in Saudi Arabia (Altuwaijiri et al., 2022), a coastal site on the Central Mediterranean Sea (Perrone et al., 2019), and the Sahara (Chirizzi et al., 2017), but considerably lower than those reported for mineral soil dust in Japan (23 pmol/min/ μ g) (Nishita-Hara et al., 2023). The differences in OP between the sites may be partially

attributed to varying concentrations of redox-active species. As seen in Table 1, the concentrations of redox-active metals such as Mn, Fe, and Cu in the GSL dust samples were higher compared to the concentrations in other regional playas. Several studies have reported that transition metals may play a role in triggering oxidative stress and inflammation in the lungs by catalyzing the formation of ROS through participation in Fenton reactions and generating highly reactive superoxide radicals (Jomova et al., 2023; Jomova and Valko, 2011; Kim et al., 2021; Valko et al., 2016).

3.3. Relationship between OP and elemental composition

To gain a deeper insight into the primary chemical constituents associated with the higher OP of the dust samples, the relationship between OP values and metal components in the dust samples was assessed using Pearson's r correlation coefficients. Figs. 7 and 8 show the

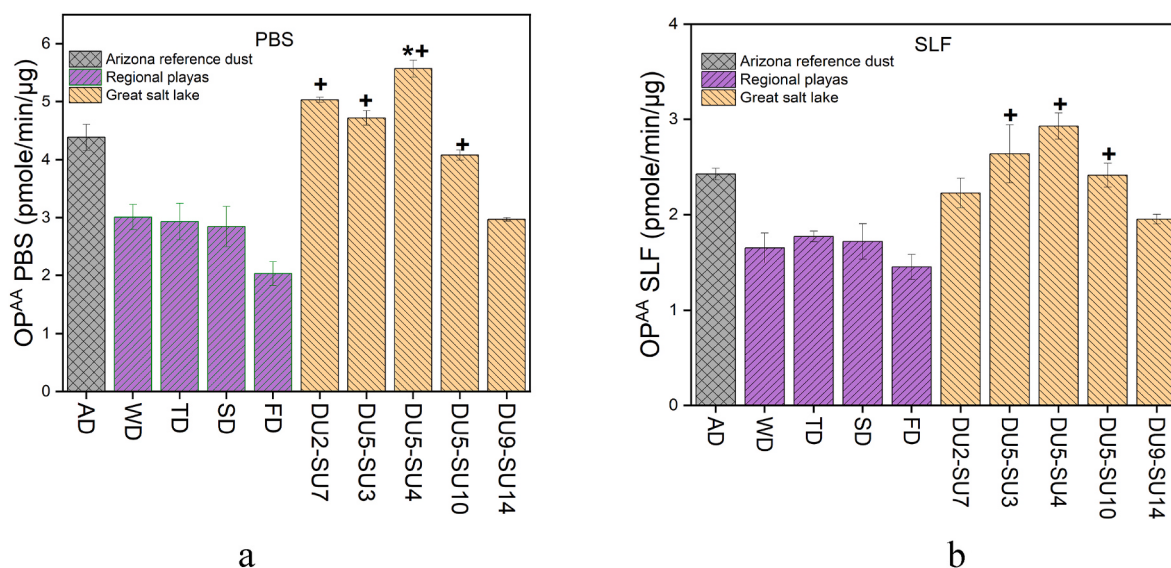


Fig. 5. OP^{AA} of the dust samples in (a) PBS and (b) SLF. Error bars represent the standard deviation of the mean from three replicate measurements. Asterisks denote significantly higher than AD (* $p < 0.05$), and plus signs denote significantly higher than other regional plays (+ $p < 0.05$).

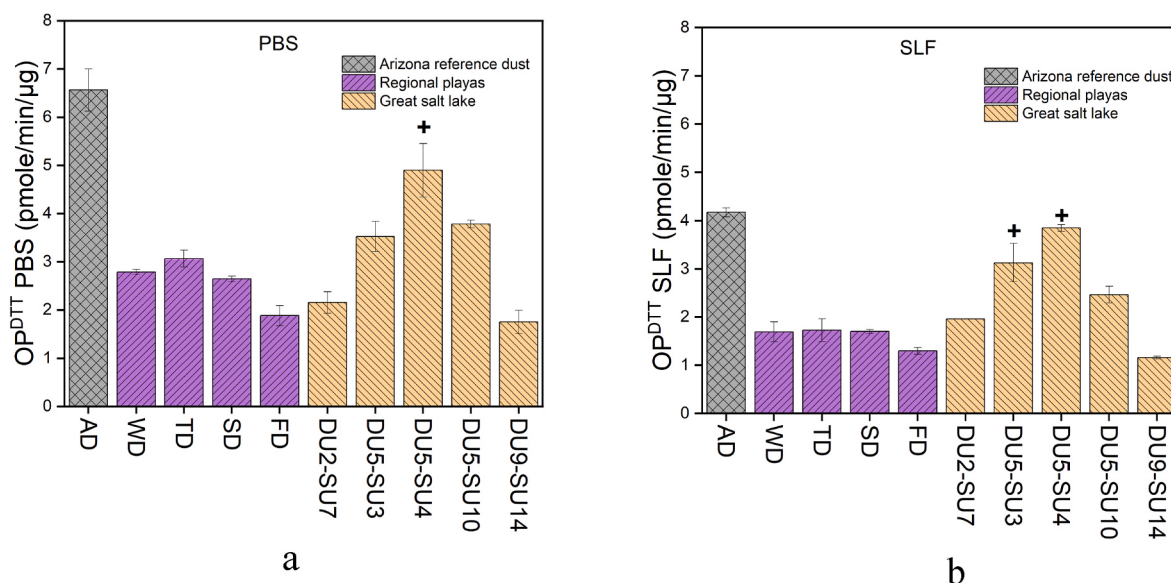


Fig. 6. OP^{DTT} of the dust samples at different sampling sites in (a) PBS and (b) SLF. Error bars represent the standard deviation of the mean from three replicate measurements, and plus signs denote Significantly higher than other regional plays (+ $p < 0.05$).

correlation coefficients between OP values and elemental concentration.

In the AA assay (Fig. 7), we observed a positive correlation between the OP^{AA} and metal concentrations for the following constituents: (OP^{AA}PBS: Pb, $r = 0.64$; Cu, $r = 0.61$; Mn, $r = 0.49$; Fe, $r = 0.43$; and Cr, $r = 0.38$) and (OP^{AA}SLF: Pb, $r = 0.53$; Mn, $r = 0.52$; Cu, $r = 0.5$; Fe, $r = 0.47$; and Cr, $r = 0.4$). For the DTT assay (Fig. 8), the correlation coefficients between the OP (OP^{DTT}-PBS and OP^{DTT}-SLF) and metal concentrations were positive for (OP^{DTT}PBS: Al, $r = 0.46$; Mn, $r = 0.4$; Fe, $r = 0.4$; Cu, $r = 0.39$; and Cr, $r = 0.39$) and (OP^{DTT}SLF: Fe, $r = 0.48$; Cu, $r = 0.45$; Cr, $r = 0.45$; Mn, $r = 0.44$; and Al, $r = 0.31$). These results suggest an association between the redox activity of these metals in the dust samples to generate ROS. Previous studies on the OP of PM₁₀ have linked AA and DTT to redox-active transition metals, especially Cu and Mn (Malakootian et al., 2022; Pietrogrande et al., 2018). The relationship between OP values and metal components in the dust samples was also assessed using principal component analysis (PCA) factors. The first two principal components combined explained about 55–57 % of the

total variance in the data set in the OP^{AA} and OP^{DTT} assays, respectively. Fig. S2 shows the relationship between metal composition and OP in the OP^{AA}-PBS assay. The component space (Fig. S2) indicates that PC1 accounts for 34% of the total variability in the dataset. PC2 follows PC1, explaining approximately 23% of the total variance. Notably, Fe, Cu, Cr, Mn, and Pb are clustered in the same quadrant as the OP, indicating their correlation and relative distance to the OP variable compared to other elemental metals. This correlation pattern extends similarly across other assays (Figs. S2–S5), as shown in the supplementary material.

3.4. Effect of assay and solvent choice on OP

Figs. 9 and 10 show the correlation between OP values for the two OP assays (AA and DTT) and two solvents (PBS and SLF). The OP assays (AA and DTT) display different sensitivity toward redox-active species in the dust samples (Fig. 9). The correlation between OP^{AA} PBS and OP^{DTT} PBS was 0.56, and the correlation between OP^{AA} SLF and OP^{DTT} SLF was

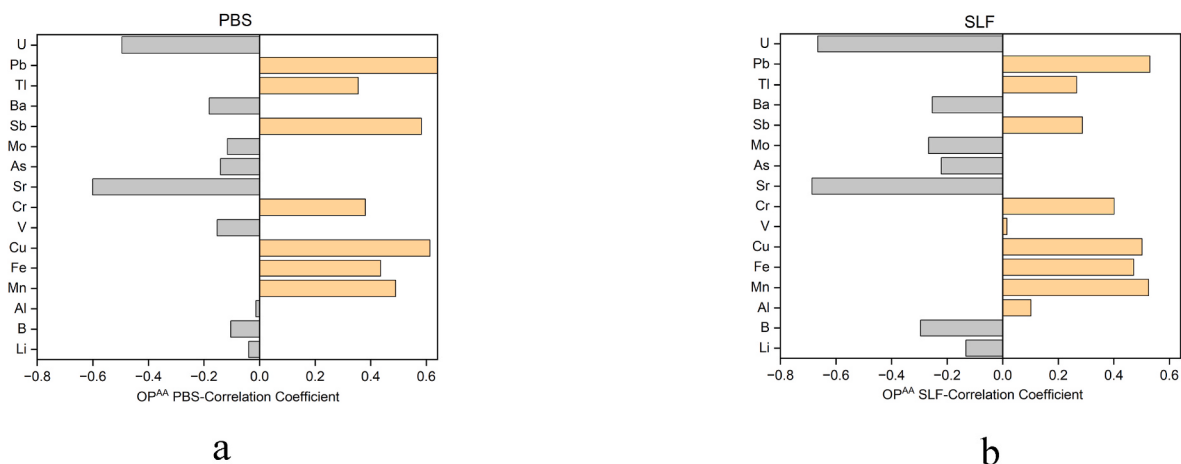


Fig. 7. Bar chart of the correlation coefficients between the OP^{AA} (OP^{AA} dust samples suspended in (a) PBS and (b) SLF) and metal concentration of all dust samples (GSL, regional playas, and AD).

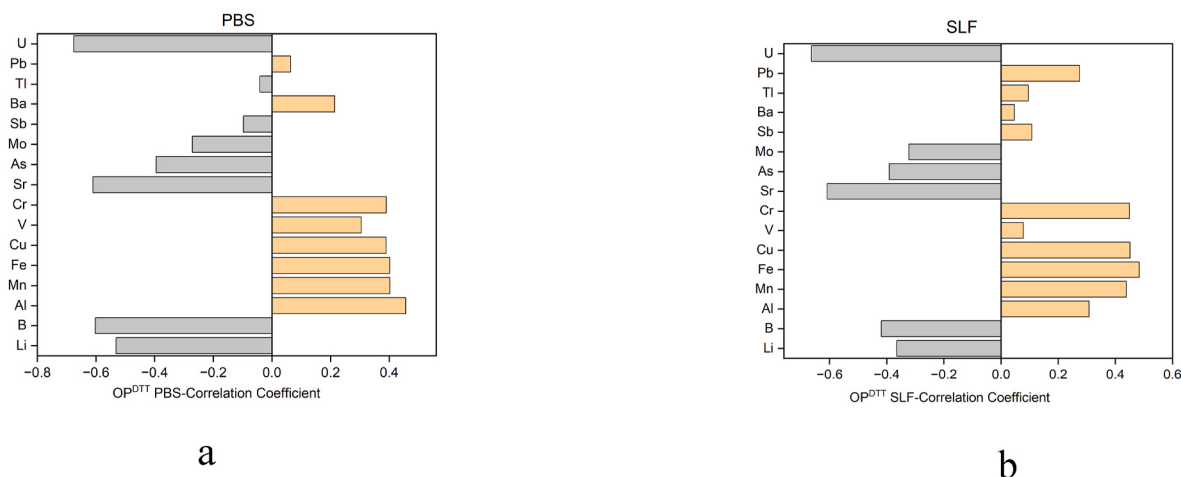


Fig. 8. Bar chart of the correlation coefficient between the OP^{DTT} (OP^{DTT} dust samples suspended in (a) PBS and (b) SLF) and metal concentration of all dust samples (GSL, regional playas, and AD).

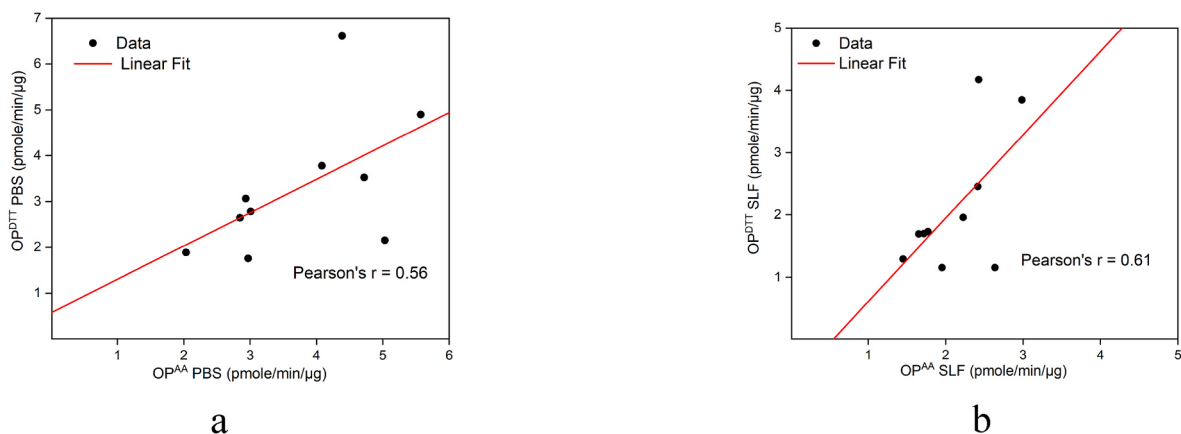


Fig. 9. Correlation between OP assays for all the dust samples (GSL, regional playas, and AD).

0.61. This indicates that the two OP assays (AA and DTT) display different sensitivity toward redox-active species in the dust samples suspended in the solvent. Figs. 5 and 6 show that the OP^{AA} for each sample was higher than the corresponding OP^{DTT} for each solvent except for the AD sample, most likely because the AA assay was more sensitive

to redox-active metals in the dust samples. The OP^{AA} assay displayed sensitivity towards Pb, Cu, and Mn in both solvents, while the OP^{DTT} was associated with Al and Fe. OP assays exhibit distinct responses to redox-active compounds due to the unique reaction mechanisms underlying each assay (Bates et al., 2019; Clemente et al., 2023; Visentin et al.,

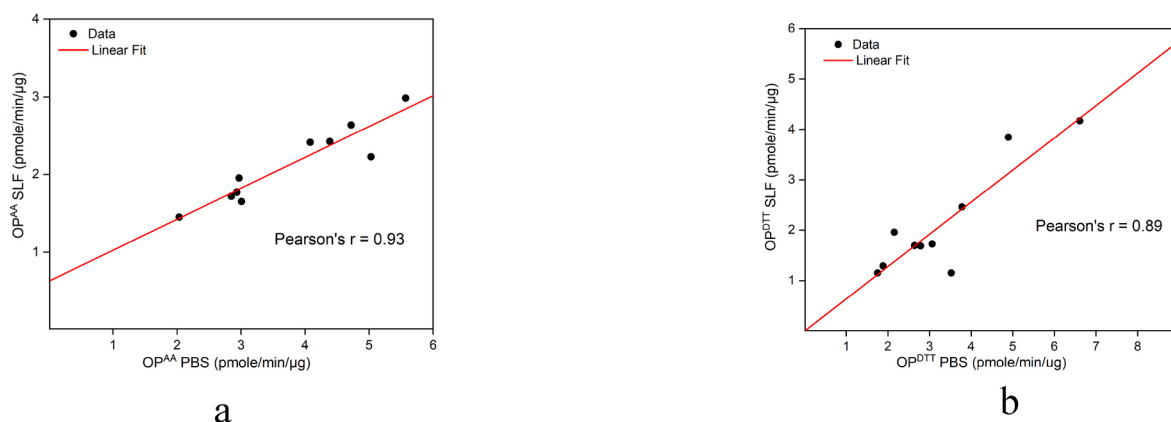


Fig. 10. Correlation between OP solvents for all the dust samples (GSL, regional playas, and AD).

2016a). The findings align with some previous studies (Bates et al., 2019; Clemente et al., 2023; Perrone et al., 2019; Shahpoury et al., 2022) but contradict others that have observed similar sensitivity of both assays to the same redox-active species (Pietrogrande et al., 2019; Visentin et al., 2016b). Although there is no consensus on the most appropriate assay for accessing the OP of PM (Ayres et al., 2008; Calas et al., 2018; Sauvain et al., 2013), comparing different acellular OP measurements for a given set of ambient PM samples is important because the assays could potentially provide complementary information on the OP of the PM. This information can also give insight into the chemical composition responsible for the assay responses, facilitating further research into health effects and the development of more effective control strategies.

Similar to the choice of assay, the values of OP also differ based on the choice of solvent (Fig. 10). Although the two solvents exhibited similar trends in both OP assays ($OP^{AA} r = 0.93$ and $OP^{DTT} r = 0.89$), overall, the OP-PBS of most of the dust samples were significantly higher than the OP-SLF ($P < 0.05$) for both OP assays (Fig. S1). Such differences may be mainly ascribed to variations in soluble fractions of redox-active species in the two solvents. Our findings are consistent with several literature papers that reported high redox activity in PBS compared to SLF (Cigánková et al., 2021; Expósito et al., 2023; Pietrogrande et al., 2021). Pietrogrande et al. observed that the $OP^{AA}PBS$ was nearly double that of $OP^{AA}SLF$ and methanol extracts. Aside from the differences in solubility, the reduction of OP in SLF compared to PBS may also be explained by the formation of metal complexes in SLF that decrease antioxidant oxidation. Previous studies have suggested that chloride compounds play a role in reducing the redox activity of metals in AA solution (Cigánková et al., 2021; Expósito et al., 2023; Murekhina et al., 2022). Murekhina et al. (2022) proposed that the reduction of AA oxidation in SLF was due to the formation of Cu (I) and Cu (II) complexes when chlorides in SLF react with Cu. These complexes exhibit distinct oxidation rates compared to the unbound cation.

4. Conclusions

The study investigated the OP (using the AA and DTT assays) and elemental composition of PM_{10} dust from the GSL, other regional playas, and a reference AD. Our results suggest that GSL dust PM_{10} generally exhibits greater OP than other regional playas, which is potentially concerning for the residents who live adjacent to the GSL lakebed. We also found that the concentration of As in all the GSL dust samples exceeded the EPA soil residential RSL, and the concentrations of Li in some of the GSL dust samples exceeded the EPA soil residential RSL. Our statistical analysis (PCA coupled with correlation analysis) indicated that metals such as Pb, Cu, Mn, Fe, Cr, and Al were associated with high OP of the GSL dust. This study showed that the choice of assay and extraction solvent affects the OP activity of redox-active fractions of

PM_{10} and highlights the importance of using different OP assays to investigate the sensitivity of redox-active species towards antioxidant surrogates.

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CRediT authorship contribution statement

Reuben Attah: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Kamaljeet Kaur:** Writing – review & editing, Methodology, Investigation, Formal analysis. **Kevin D. Perry:** Writing – review & editing, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Diego P. Fernandez:** Writing – review & editing, Methodology, Investigation. **Kerry E. Kelly:** Writing – review & editing, Supervision, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.atmosenv.2024.120728>.

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