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Effectiveness of gravity based particle separation and soil washing for reduction of Pb in a clay loam shooting range soil

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ABSTRACT

A heavy particle concentrator (HPC), which can separate heavy metal particles from soil based on density, was examined for remediation of shooting range soil contaminated with lead (Pb) from a military training area. Concentrations of Pb in the stockpiles ranged between 1403 and 4300 mg/kg. The soils had high clay and silt content and were found to have relatively high exchangeable Pb, between 238 and 1480 mg/kg. After initial treatment by HPC, total Pb in the soil was reduced by 28%–56%. The fine soil fraction (<250 μm) was found to still have relatively high Pb after HPC treatment. A greater removal efficiency was achieved by passing contaminated soil through the HPC a second time.

Scanning electron microscopy (SEM) analysis of the treated stockpile indicated Pb present in the soil sorbed on soil particles or as very fine discrete particles (<10 - 20 μm) after initial HPC treatment. The addition of a chemical washing agent, ethylenediamine-N, N'-disuccinic acid (EDDS) was effective for removal of 68% of residual Pb after a single HPC treatment and may be considered for high clay soils.

1. Introduction

Shooting ranges may contain a significant burden of Pb in soil, with annual deposition of up to 800 kg reported for a single range (Knechtenhofer et al., 2003). Shooting ranges are operated throughout the world for military training and recreation, with shooting activities adding potentially large volumes of Pb-based bullets to the soil (Ahmad et al., 2012; Darling and Thomas, 2003; Sanderson et al., 2012; Sehube et al., 2017; Sorvari et al., 2006). A report by Myhre et al. (2013) estimated annual Pb deposition from ammunition of up to 103 metric tons in the Norwegian Defence sector.

At rifle ranges, these bullets are subject to fragmentation upon impact with the soil berm, which captures the fired bullets. Fragmentation further increases as bullets impact with bullet fragments contained in the berm soil (Hardison et al., 2004). The bullets in berm soil also undergo weathering over time releasing Pb and co-contaminants (Sb, Cu and As) into the soil, which may leaching down the soil profile and surface runoff into nearby water ways (Etim, 2016; Mariussen et al., 2015). Weathering in the soil is dependent on soil physico-chemistry and is affected by soil texture (sand, clay, silt), soil pH, soil moisture, organic matter temperature and humidity (Sanderson et al., 2018). Less weathering has been reported

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in sands with low organic content due to lower water holding capacity (soil moisture) and CO_2 in soil (Liu et al., 2013; Yin et al., 2010). Low soil pH, organic matter and soil moisture promote weathering in the soil (Ma et al., 2007; McLaren et al., 2009). Contamination of soil by Pb from shooting range soil is also a concern for ecotoxicity (Rodríguez-Seijo et al., 2017; Sanderson et al., 2014).

Various management and remediation strategies have been applied for treatment of shooting ranges. These approaches broadly fit into three categories 'mobilisation', 'stabilisation' and 'removal' (Sanderson et al., 2018). Examination of amendments for stabilisation of Pb and associated contaminants in shooting ranges has been an ongoing area of research interest (Sanderson et al., 2018; Seshadri et al., 2017; Tandy et al., 2017; Yan et al., 2016). A number of papers have examined mobilisation for treatment of contaminants in shooting range soils including phytoextraction (Bandara and Vithanage, 2016; Rodríguez-Seijo et al., 2016), soil washing (Etim, 2017; Lafond et al., 2014; Yoo et al., 2016) and electrokinetic applications (Pedersen et al., 2018). Despite the relative effectiveness of these methods consideration must be given to potential ongoing release of metals from bullets and fragments remaining in the soil, which will not be completely addressed by 'mobilisation' and 'stabilisation' methods, which primarily deal with the fraction of Pb associated with the soil. For example, Lafond et al. (2014) and Yoo et al. (2016) examined the < 250 and < 75 μm respectively.

Removal refers to approaches involving the physical separation of Pb bullets from the soil, for the management and remediation of Pb. Physical separation may encompass several different processes including mechanical screening, froth flotation, hydroclassification, attrition scrubbing, gravity concentration, magnetic separation and electrostatic separation (Dermont et al., 2008). These technologies are effective to varying degrees for removal of metals (Dermont et al., 2008). Periodic removal from the soil berm is recommended as part of range maintenance to reduce fragmentation of bullets from bullet on bullet impact and the environmental risk from Pb burden (USEPA, 2001).

The method of separation by mechanical screening requires caution. Mechanical screening typically removes the coarse fraction > 4 mm, but fine Pb fragments remain and substantial Pb is found in the > 2 mm fraction of shooting range soils (Sanderson et al., 2012). Mechanical screening may also transfer fine Pb fragments to the soil by the abrasive action (Yin et al., 2010). Additionally, Pb and other co-contaminants may also be sorbed to the soil after weathering and release from Pb-bullets, particularly due to the high clay content and alkaline pH of the soil (Bradl, 2004).

Gravity based particle separation methods have potential for removal of Pb bullets and fragments from shooting range contaminated soil and significantly reducing the Pb burden of soil by producing a small volume of Pb concentrate (Marino et al., 1997; Hintikka et al., 2001). However, while typical hydroclassifiers and gravity concentrators (jig, shaking table and spiral) have a good applicability for the sand fraction they may not be suitable for fine particles (> 63 μm) (Dermont et al., 2008). The effectiveness of a heavy particle concentrator (HPC), which is used in the mineral industry for separation of metal bearing particles based on density was investigated for separation of Pb from a soil with high clay content, which may sequester Pb in the fine soil fractions. Scanning electron microscopy (SEM) and chemical analysis was applied to provide insight into the effectiveness of Pb separation.

2. Methods

A previous investigation optimised the parameters of the HPC for treatment of stockpiled contaminated soils from a shooting range at the Mount Sturt training area (MSTA) in Townsville, Queensland, Australia (Thangavadivel et al., 2018). The feed conveyor, trommel and HPC belt speed were set at 1500, 1980 and 4 rpm respectively and the HPC inclination angle was 4°, material distribution chute extension 100 mm, and water flow 480 l/min based on optimisation and treatment trials (Thangavadivel et al., 2018). This study examined the contaminated Pb stockpiles at the MTSA in Townsville to determine effectiveness of the HPC for removal of Pb from the soil.

The HPC treatment process outlined in Fig. 1 consisted of stockpiled soil being fed through 40 mm screen and hopper, which is conveyed to the trommel scrubber. The soil is scrubbed and screened in the trommel, with oversize material discharged and remaining material (> 15 mm) washed through the trommel screen to the distribution shoot and heavy particle concentrator belt. The concentrator belt separates heavy and light particles by material fluidisation. The heavy particle concentrate including bullets and metallic fragments were collected for recycling. The fines suspended in water in the light particle fraction flowed with the water to a lined tailings pond. The remaining of the soil was dewatered and stored in a separate pond for verifying residual Pb content. The HPC treated soil was re-fed through the HPC treatment process to further remove the contaminant at the same HPC operating parameters.

Portable XRF (Niton XL3T 950) was used to analyse Pb concentrations in the soil on site before and after treatment.

2.1. Laboratory analysis

Soil characterisation was performed using standard methods in the lab. Soil pH and EC were measured by 5:1 water extraction, CEC by BaCl2 method, organic matter by wet oxidation and soil texture by hydrometer method. Exchangeable Pb was determined by 0.1 M BaCl2 extraction. Total metals, including analysis by soil size fraction (> 2 mm–500 μm, $>$ 500–250 μm and > 250 μm fractions) were analysed in the lab by microwave assisted digestion by USEPA 3051 method.

Ethylenediamine-N,N'-disuccinic acid (EDDS) was examined for removal of metals from HPC treated soil in batch studies. A 1:1 metal:EDDS molar ratio was added in solution to 4 g soil in centrifuge tubes. The solution:soil ratio was 5:1 (20 ml) and samples were shaken on an end over end shaker for 12 h and then centrifuged at 3000 rpm for 10 min prior to collecting a subsample of the solution for analysis.

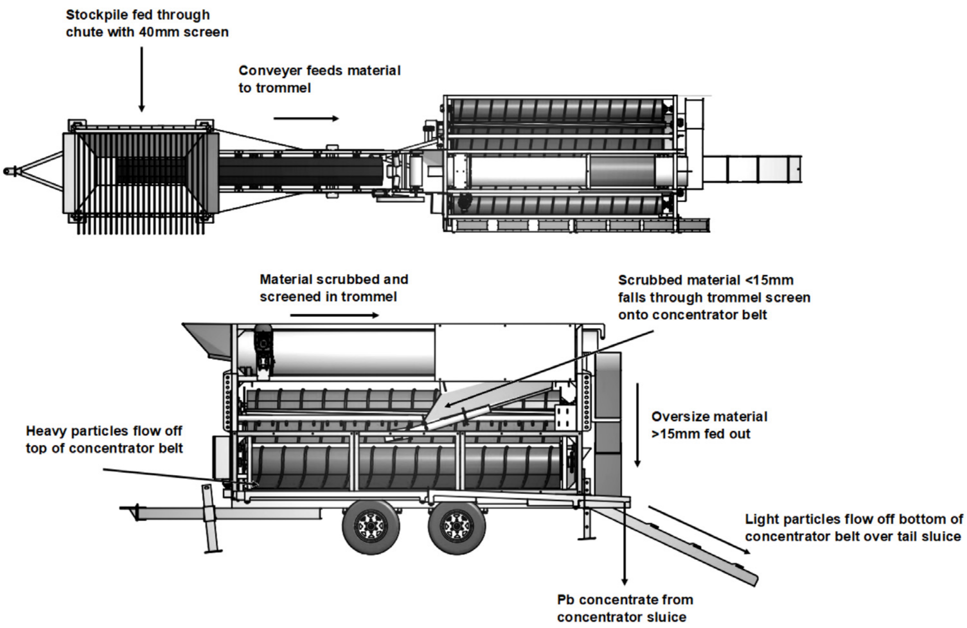

Fig. 1. Schematic of Pb separation process steps within the heavy particle concentrator (HPC).

Table 1
Soil properties of the shooting range stockpiles.

	pH	EC (µs/cm)	CEC cmol/kg	%clay	%silt	Sand %	Org%
Stockpile 1	8.53	89.1	15.1	29.9	30	40.1	0.41
Stockpile 2	8.03	78	9.05	14.23	23.95	61.82	
Stockpile 3	6.88	216	10.05	14.23	29.95	55.82	

Table 2
Total metals in the shooting range stockpiles.

mg/kg	Pb	Cu	Sb	As
Stockpile 1	3265 ± 502	268 ± 130	3 ± 1.4	4.5 ± 0.71
Stockpile 2	4300 ± 823	140.87 ± 27.6	4.47 ± -	5.67 ± -
Stockpile 3	1403 ± 490	12.70 ± -	BD ±	1.09 ± -

Bioaccessibility of Pb was determined using the SBRC-G method, a chemically simulated gastric extraction (Juhasz et al., 2009). Briefly a solution was prepared with 0.4 M Glycine and adjusted to pH 1.5 with HCl. The solution was heated to 37 °C in a temperature controlled room and stockpile samples were extracted for 1 h at a 1:100 soil:solution ratio in the temperature controlled room, using an end over end shaker. After 1 h the pH was checked to confirm it remained within 0.5 units as required by the procedure, and then samples were centrifuged at 3000 rpm for 10 min prior to collecting a subsample of the solution for analysis.

Chemical analysis for metals was conducted using Agilent ICPMS 7900 and Perkin Elmer AVIO 500 ICP OES. Scanning electron microscopy (SEM) (Philips XL30 SEM + Oxford ISIS EDS) was conducted on soil samples sieved to > 2 mm and > 53 µm. Imaging was conducted at 20 kV.

3. Results and discussion

3.1. Characterisation of stockpiles

Soil properties of the > 2 mm fraction are given in Table 1. The soils are sandy loam to sandy clay loam, neutral to slightly alkaline, with moderate CEC. The stockpiled soils all had elevated levels of Pb as a result of the shooting activities, with Pb in the > 2 mm fraction ranging from 1403 to 5177 mg/kg (Table 2). Copper concentration in the soil was also elevated, but the other co-contaminants of Pb (Sb and As) were relatively low.

Due to the soil clay content, CEC and alkaline pH, a proportion of Pb can be expected to be sorbed to the soil. Exchangeable Pb in the stockpiles was 23% (S1), 31% (S2) and 17% (S3) respectively (Table 3).

Table 3
Exchangeable metals in the shooting range stockpiles.

mg/kg	Pb	Cu	Sb	As
Stockpile 1	751.79 ± 519	15.2 ± 2.6	1.22 ± 0.7	0.22 ± 0.05
Stockpile 2	1480 ± 520	53.5 ± 6.9	1.73 ± 0.4	0.29 ± 0.15
Stockpile 3	238.40 ± 26	16.7 ± 0.8	0.68 ± 0.1	0.10 ± 0.01

Table 4
Pb (mg/kg) in Stockpiled and HPC treated stockpiles.

	Feed	Treated	Oversize	Tailings	Treated-Reprocessed
Stockpile 1	3265 ± 502	1695 ± 163	959[a]	2466[a]	832[a]
Stockpile 2	4300 ± 823	2476 ± 395	834 ± 37	2662 ± 1524	–
Stockpile 3	1403 ± 490	390.31 ± 181	83.53 ± 1	237.82 ± 14	262[a]

[a] XRF field readings from (Thangavadivel et al., 2018).

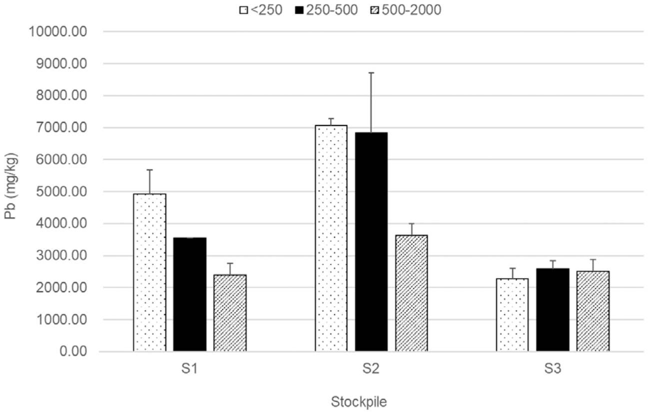

Fig. 2. Size fraction (μm) distribution of Pb in stockpiles.

3.2. HPC treatment

Stockpiled soil was fed into the treatment train as in Fig. 1. The feed soil consisted of rocks, gravel, sand, silt, clay, and organic matter, which was passed through > 40 mm screen. The screened material is passed to the trommel, which breaks clay lumps and disperses feed soil into a slurry with water to enable effective separation of Pb, particularly fine Pb particles. Due to the high clay content (up to 30%) in soil, a lower soil to water slurry ratio in the trommel was found to be beneficial to reduce the viscosity and achieve better separation of fine Pb particles. Liberation of Pb from the soil particles to discrete Pb by means such as attrition scrubbing is necessary for successful physical separation (Dermont et al., 2008; Marino et al., 1997).

The material is scrubbed and screened in the trommel with oversize material (>15 mm) being fed out, and > 15 mm passing to the concentrator belt. The concentrator belt separates particles by density, resulting in a Pb concentrate separated from the less dense soil particles (Fig. 1). The processing of soil through the HPC was up to 6.5 tonnes/hr, with Pb concentrate 26.4 – 141.60 kg/hr and resulting treated soil on average 2000 kg/hr (> 15 mm).

The concentration of Pb in the stockpiled soil was reduced after undergoing initial separation by HPC. Treatment of stockpiles resulted in reduction of Pb by 42%–72% in the treated soil (Table 4). HPC screened particles (>15 mm) consisting of stones and clay clods contained Pb up to 959 mg/kg. The tailings, which consisted of fines (clay and silt) not removed during dewatering contained up to 2662 mg/kg Pb. The tailings and oversize particles were retained for later treatment with rock phosphate for reduction of residual leachable Pb.

Reprocessing of stockpile 1 and stockpile 3 treated soils decreased Pb in the treated soil by a further 33%–51%, for total Pb removal between 74%–81%. This was likely by greater liberation of Pb bound to soil. The Pb in soil was reduced to below the National Environmental Protection Measure (NEPC, 1999) health investigation level for commercial industrial land (HIL D) of 1500 mg/kg Pb.

Stockpile 2 which had a high percentage of Pb remaining was investigated using SEM.

3.3. Soil size fractionation and bioaccessibility of Pb

Lead particle size (> 2 mm – 500 μm, > 500 to 250 μm and > 250 μm fractions) was examined in stockpiles and a subset of before and after treatment samples (> 250 μm fraction). The fractionation of Pb is presented in Fig. 2. The lead

Table 5
Pb in < 250 µm soil size fraction after single HPC treatment.

	Pb (mg/kg)
Stockpile 2 untreated	7064
Stockpile 2 treated	4658

Table 6
Removal of Pb from HPC treated Stockpile 2 material by EDDS.

	Total mg/kg	Removed by EDDS (mg/kg)	Residual (mg/kg)	%
Treated Soil	2662.7	1829.1	833.6	68.7
Oversize (>15mm)	833.8	568.4	265.4	68.2
Tailings	2476	1693.5	782.5	68.4

in the stockpiles of the $>$ 2 mm fraction was concentrated in the finer fractions on a per mass basis. Intact bullets and larger fragments will be readily separated from soil, but in the fine soil fraction Pb may be associated with the soil. Soil passed through the HPC still contained high levels of Pb in the $>$ 250 µm fraction (≈5000 mg/kg), though less than soil untreated by HPC (≈7000 mg/kg) (Table 5). Additional HPC processing may be able to reduce Pb in this size fraction.

Previous investigations of soil size fractionation of Pb have found Pb concentrated in the finer soil fractions (Fayiga et al., 2011; Sanderson et al., 2012). Other researchers have found Pb more concentrated in coarser fractions due to metallic fragments, though Pb in the fine fractions is still elevated (Dermatas and Chrysochoou, 2007).

Bioaccessibility was determined for stockpile soils, including for before and after treatment for selected samples. Bioaccessibility of Pb was reported between 90 and 100% for all stockpile samples (data not shown). In treated soils the bioaccessibility of residual Pb remained high $>$ 90%. Previously the bioaccessibility of shooting range Pb has been found to be similarly high (between 90%–100%) (Bannon et al., 2009; Sanderson et al., 2012).

3.4. SEM investigation of the nature of Pb in MSTA stockpile soil

The soil was expected to have strong ability to sorb Pb (due to clay content and alkaline pH). This was affirmed by the relatively high proportion of exchangeable Pb reported for the soil. SEM was used to examine residual Pb in soils after initial HPC treatment. SEM showed agglomeration of precipitated Pb on the surface of soil particles (∼> 10 µm – 200 µm in size) (Fig. 3) and also discrete Pb particles (1 – 10 µm in size) (Fig. 4) after initial HPC treatment.

This Pb that is sorbed to the soil may not be as readily removed by physical separation as coarse discrete metallic phases. However further dilution of soil slurry and higher speed and greater agitation of trommel may help to disperse clay and aid separation of Pb. As previously noted reprocessing of treated soil was successful in reducing the Pb burden of the soil. Other methods may be considered for enhancing the removal of Pb during the HPC separation process, such as the incorporation of a chemical washing agent.

3.5. Soil washing

The use of a chelating agent was examined in a batch study to determine the effect of a washing agent on removal of residual Pb associated with the soil after HPC treatment (Table 6). The biodegradable chelate EDDS was chosen as it has been demonstrated to extract Pb from shooting ranges associated with the exchangeable fraction (Sanderson et al., 2017) The use of EDDS further reduced Pb in HPC treated soils, oversize particles and tailings by around 68% (Table 6). The amount of Pb removed by EDDS was comparable to the exchangeable fraction of the soil (1480 ± 520, Table 2). The treatment was applied in a 1:1 chelate to metal molar ratio. A greater removal efficiency may be obtained when using higher chelate doses.

The lab trial suggests a chemical washing agent may be added to HPC treatment train to increase removal of Pb, particularly from soil with high clay content.

4. Conclusion

Gravity based separation is a potential means of effectively remediating lead in shooting range soils as bullets and bullet fragments would be readily removed by such a method.

However, in high clay and silt containing soils there may be high levels of Pb bound to the soil by sorption, as Pb is released over time by the weathering of bullets and bullet fragments.

The effectiveness of a heavy particle concentrator was tested to determine the ability to separate Pb from soil and reduce total metal loading in shooting range soils. This study confirmed Pb was bound to the soil and found the operating parameters to obtain dispersal of clay and silt in the slurry were important for successful treatment. A second processing with greater water volume was able to achieve further reduction of Pb in the soil and chemical soil washing was also

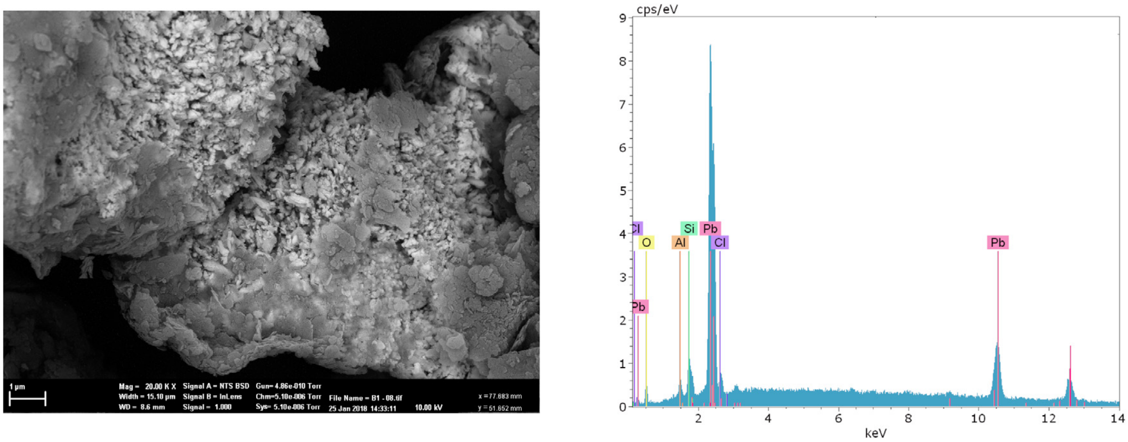

Fig. 3. SEM backscatter image and associated EDS spectra of Pb sorbed on soil after HPC treatment.

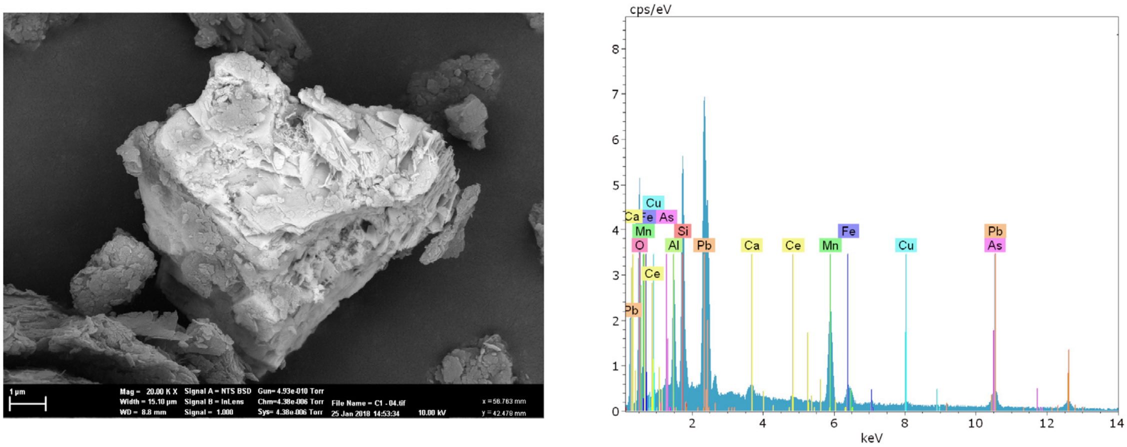

Fig. 4. SEM backscatter image and associated EDS spectra of discrete Pb particle in soil after HPC treatment.

shown to be potentially effective as part of this process. Subsequent treatment by chemical stabilisation of the residual may be required, particularly for tailings material.

The advantage of the technology is the removal of a significant proportion of bullets and fragments from the soil, reducing the potential for environmental impact from weathering over time. It also reduces abrasion and release of fine Pb that may occur with mechanical separation. The collected Pb is able to be recycled offsetting costs and increasing sustainability of this remediation approach. Therefore this remediation technique should be considered as a potential management strategy for Pb in shooting ranges.

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.

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