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Mineral Signatures in Oued K'sob River (Algeria): Highlighting Geological Variations and Potential Anthropogenic Impacts

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Abstract: This study examines the mineral composition of sediment samples collected from five distinct zones along the Oued K'sob River in Bordj Bou Arreridj, Algeria using scanning electron microscopy (SEM). The analysis revealed intriguing patterns, the Zone 5 emerged as a distinct outlier, exhibiting significantly lower calcium (29.32% vs. 53.25% in Zone 1) and iron (14.94% vs. 6.31% in Zone 1) content compared to other zones, while displaying a higher silicon content (28.82% vs. 25.27% in Zone 1). This suggests a unique geological origin or specific weathering processes shaping the mineral fingerprint of Zone 5. Interestingly, Zones 1, 2, and 3 displayed a relative consistency in silicon content (around 25%, with pairwise comparisons exceeding $p > 0.05$), suggesting potential similarities in mineral sources or enrichment mechanisms within their watersheds. However, Zone 4 deviated slightly, showing a higher calcium content (64.18% vs. 29.32% in Zone 5) and lower silicon content (17.99% vs. 28.82% in Zone 5), indicating potential differences in its geological makeup or weathering processes. Furthermore, all zones exhibited trace amounts of elements like lead (Pb) and cadmium (Cd) (ranging from 0.76% to 2.16% for Pb and 0.000% to 0.114% for Cd) (Please recheck these values They are very high). This warrants further investigation to understand their potential anthropogenic origins and impact on the river ecosystem. Further investigations incorporating detailed geological surveys, isotopic analyses, and expanded sampling strategies are recommended to gain a more comprehensive understanding of the complex factors shaping the sediment chemistry within the Oued K'sob River system.

Key words: Oued K'sob River, sediment chemistry, mineral composition, trace elements, silicon, calcium, iron.

The waters of wadis (ephemeral streams) and even groundwater is increasingly susceptible to contamination from various sources. These include natural and industrial organic and mineral compounds, plant debris, waste from human activities, and toxic elements originating from industrial processes (Aziz *et al.*, 2023). Pollution by heavy metals is a growing environmental concern, caused by a combination of natural leaching from geological formations and anthropogenic activities such as industrial discharge and agricultural practices (Arora *et al.*, 2023). This contamination has led to the contamination of thousands of hectares of arable land, posing a significant threat to human health (Perković *et al.*, 2022).

The toxicity of urban and industrial wastewater can be attributed to the presence of heavy metals, chemicals, and other products resulting from chemical reactions occurring within wastewater pipes. The Bordj Bou Arreridj region in Algeria is one such area experiencing industrial pollution. This issue has become particularly worrisome in the past decade due to the region's rapid industrial development, especially in sectors like electronics, appliances, and textiles (Khelili *et al.*, 2022).

Oued K'sob, a vital watercourse in the region, acts as a final collector for all wadis in Bordj Bou Arreridj. Located south of the wilaya (province), it flows past both the wastewater treatment plant that discharges treated water into the wadi and the industrial zone that directly releases untreated wastewater into the stream.

Phytoremediation is a green technology that utilizes plants and their associated microbes to decontaminate polluted environments. Species of Typha, such as *Typha latifolia* and *Typha domingensis*, are particularly promising candidates for this process (Ebrahimbabaie *et al.*, 2023; Martínez-Martínez *et al.*, 2023; Qays *et al.*, 2023).

Materials and Methods

Study area, water and soil sampling

Bordj Bou Arreridj, a significant urban center, faces severe environmental challenges due to the discharge of urban, agricultural, and industrial effluents into the Oued K'sob River (Fig. 1). Originating in the Sétifian High Plateaus and covering an area of 1484 km², the river, with an average slope of 9.3%, culminates in the K'sob dam located in the M'sila region. Once a hub for artisanal fishing, the Oued K'sob is now heavily polluted by industrial activities. The indiscriminate establishment of industrial zones, primarily along the riverbank, has exacerbated the situation (Benkadja *et al.*, 2013).

The region's industrial landscape is diverse, encompassing electronics, paper, textiles, plastics, agri-food, building materials, metals, chemicals, and cosmetics. Notable industries include Condor, Géant, Upac-TCL, EMBAG, Lafarge, SMMARTEX, SAIM-TEXTILES, GOOD-NIGHT, Atia Electronic, Plast Kammar, Plastbros, Sarl Alsan Plastic, Sarl ALGM, Spa creative invest (CI), Sarl Maghreb plastique, Sarl GIPATES, Sarl MGI, Sarl Torche, Biscostar, Bellih, Falco, Zouaoui, SNC Belarbi, Agglo BBA, SNC FZI, SARL béton Salsabil, Argilor, Sarl MNTB, Sarl ZAS, Sarl MCA, Sarl Agrobba, IRRAGRIS-ANABIB, SNC Anwar et fils, Sarl Transfil, Sarl Benchagra, Ramix, Grafil, Hamoudi, Sapeint, Sarl LARC, and Ines Cosmetics. Most of these industries discharge their untreated effluents directly

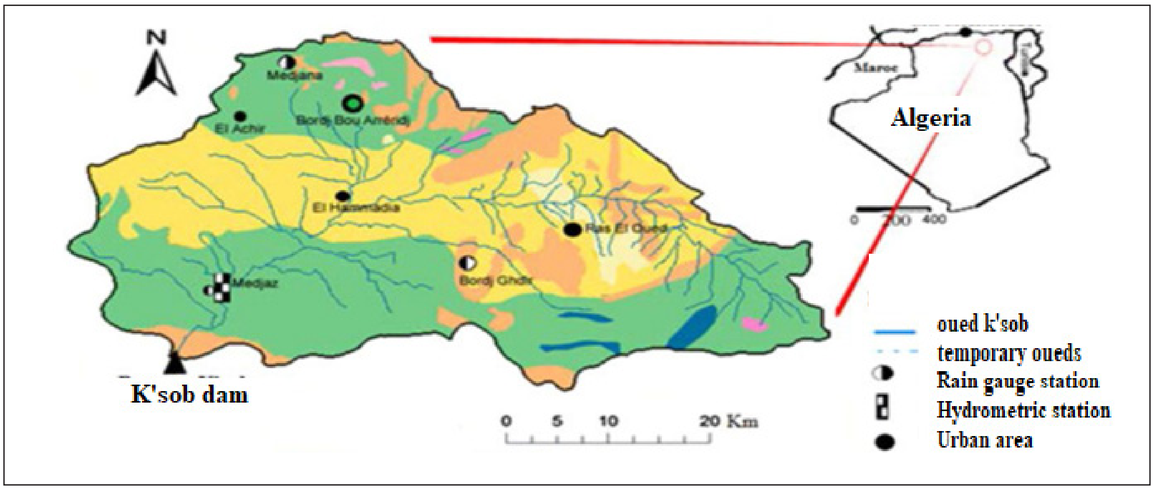

Fig. 1. Map of the watershed of the Oued K'sob (Benkadja et al., 2013).

into the Oued K'sob, while urban wastewater undergoes treatment before release.

Sampling

The sampling was carried out at five stations located 2 km apart. The first station is the direct discharge of the industrial zone, the second is located at the outlet of the wastewater treatment plant (downstream) which is located at the exit of the city of Bordj Bou Arreridj, in which the urban and industrial wastewater of the city is discharged. The third and fourth stations are located on the road to M'sila and which confine between them the discharge points of Oued Medjana and Oued Boumergued respectively. The last station is located near the commune of Elhamadia on the road to M'sila which receives the discharge of Oued El Faregh or Biata during the rainy season. Soil samples (500 g) from each point were taken in sterile bags at a depth of 50 cm to avoid any accidental surface contamination. The samples were taken 24 hours before the pre-treatment and then transported in a cooler and stored at 4°C (Bouchouata *et al.*, 2011).

Sample preparation for scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) necessitates meticulous sample preparation to ensure optimal image quality. This process involves several crucial steps:

1. Fixation: Specimens measuring 2-4 mm are immersed in a 2.5% glutaraldehyde solution at 4°C for 2-6 hours. This fixative preserves the sample's morphology and fine cellular structures.

2. Washing: Following fixation, thorough rinsing with 0.1 M phosphate buffer (three washes, 15 minutes each at 4°C) removes residual fixative and prepares the sample for subsequent steps.

3. Post-fixation: A 1% osmium tetroxide solution is applied for post-fixation (2 hours at 4°C). This step enhances the contrast of cellular membranes and organelles within the sample.

4. Dehydration: Gradual dehydration is essential for SEM imaging. The sample is subjected to a series of increasing acetone concentrations (30%, 50%, 70%, 90%, 95%, 100%) for 30 minutes each at 4°C. This removes water content and prepares the sample for microscopy. A final dehydration step with a 30% copper sulfate/70% acetone solution ensures complete moisture removal.

5. Drying: The sample can be air-dried or critical point dried. Air drying is a simpler method, but critical point drying offers superior preservation of delicate structures.

6. Mounting and Coating: The dried sample is mounted onto an aluminium stub using conductive carbon tape. To enable imaging in the SEM, a thin layer of conductive material is sputtered onto the sample surface using a sputter coater.

Important Considerations: Maintaining a cold temperature (4°C) throughout the protocol minimizes potential damage to the sample. Additionally, minimizing air exposure during transfers between solutions is crucial. After drying, the sample should be stored in a desiccator to prevent degradation from humidity fluctuations.

Results and Discussion

An analysis of the normalized weight percentage (%wt) of minerals in sediment samples from five distinct river zones yielded intriguing findings. Notably, Zone 5 demonstrated a unique mineral signature compared to other zones. It exhibited significantly lower levels of calcium (29.32% vs. 53.25% in Zone 1, $p < 0.0001$) and iron (14.94% vs. 6.31% in Zone 1, $p < 0.0001$). Conversely, a higher silicon content level was recorded (28.82% vs. 25.27% in Zone 1, $p < 0.0001$) (Table 1). This disparity suggests potential differences in underlying geological formations or weathering processes influencing Zone 5's mineral fingerprint compared to the other zones (Figures 2, 3, 4 and 5).

The normalized weight percentage (%wt) of minerals in sediment samples from five distinct fluvial zones revealed intriguing patterns, highlighting potential variations in underlying geological and environmental influences. Notably, Zone 5 emerged as a distinct outlier, displaying significantly lower calcium (29.32% vs. 53.25% in Zone 1, $p < 0.0001$) and iron content (14.94% vs. 6.31% in Zone 1, $p < 0.0001$) compared to the other zones. This stark discrepancy suggests a unique geological origin or specific weathering processes shaping the

Table 1. Normalized weight percentage (%wt) of minerals in sediment samples from five distinct river zones

wt%	Mineral composition														
	Ca	Si	Al	Fe	Pb	K	Mg	Na	Co	Zn	Cu	Ni	Mn	Cr	Cd
Zone 1	43.783	14.479	7.215	5.080	1.923	1.198	0.779	0.316	0.195	0.112	0.087	0.050	0.037	0.002	0.001
Zone 2	47.277	13.217	6.949	4.011	2.428	1.229	0.709	0.369	0.112	0.043	0.002	0.001	0.001	0.001	0.001
Zone 3	43.677	16.502	9.513	5.525	1.754	1.216	0.621	0.419	0.252	0.139	0.083	0.001	0.001	0.001	0.001
Zone 4	52.009	10.000	5.030	4.122	2.058	0.963	0.745	0.310	0.068	0.064	0.041	0.023	0.001	0.001	0.001
Zone 5	31.314	19.176	8.582	17.544	0.896	1.334	2.692	0.490	1.236	0.409	0.062	0.034	0.019	0.178	0.247

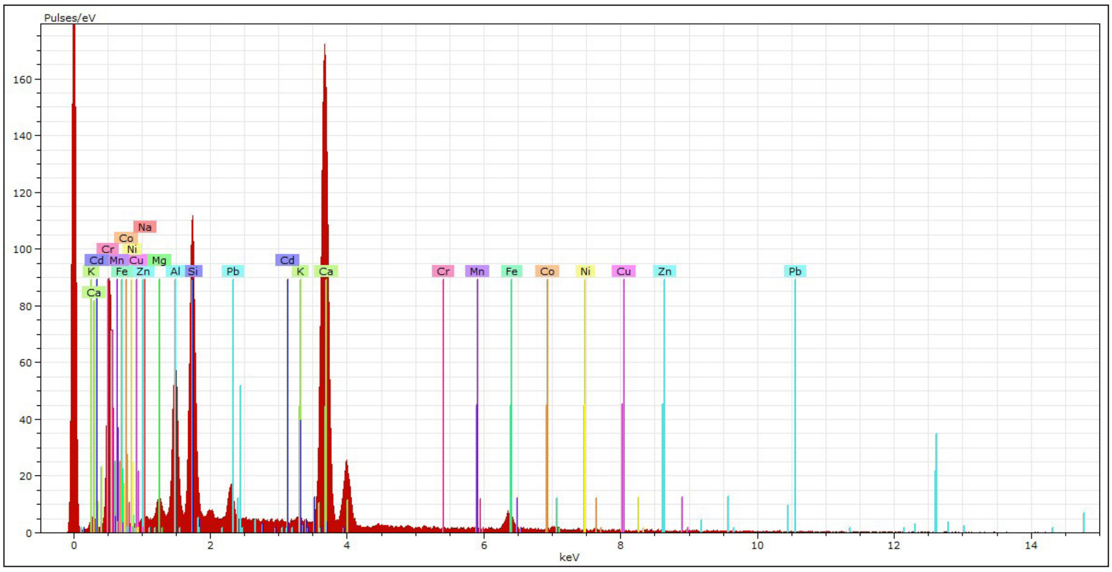

Fig. 2. SEM Mineral fingerprint Zone 1.

mineral fingerprint of Zone 5 compared to its counterparts.

Intriguingly, an analysis of silicon content revealed relative consistency across Zones 1, 2, and 3 (25.27%, 21.50%, and 22.24%, respectively), with all pairwise comparisons exceeding a p-value of 0.05. This observation suggests potential similarities in mineral sources or enrichment mechanisms within the watersheds of these zones (Figs. 2, 6, 7, 8 and 9). However, Zone 4 deviated slightly with higher calcium (64.18% vs. 29.32% in Zone 5, $p < 0.0001$) and lower silicon content (17.99% vs. 28.82% in Zone 5, $p < 0.0144$), indicating potential differences in its geological makeup or weathering processes (Table 2).

Furthermore, all zones exhibited trace amounts of elements like lead (Pb) and cadmium (Cd) (0.76-2.16% and 0.000-0.114%, respectively), highlighting the need for further investigation into potential anthropogenic influences on the mineral composition of these river systems.

The analysis of normalized atomic percentage (%At) of minerals in sediment samples from five distinct river zones revealed intriguing patterns, with Zone 5 potentially deviating from the others. This zone displayed significantly lower levels of calcium (29.32% vs. 53.25% in Zone 1, $p < 0.0001$) and iron (14.94% vs. 6.31%

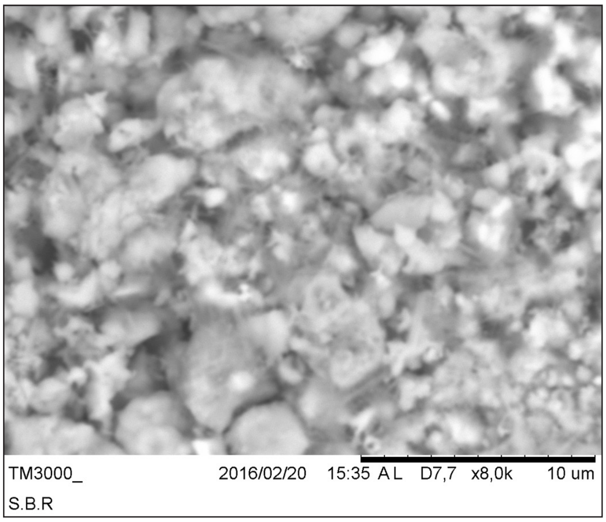

Fig. 3. SEM Image of elemental mapping (zone 1).

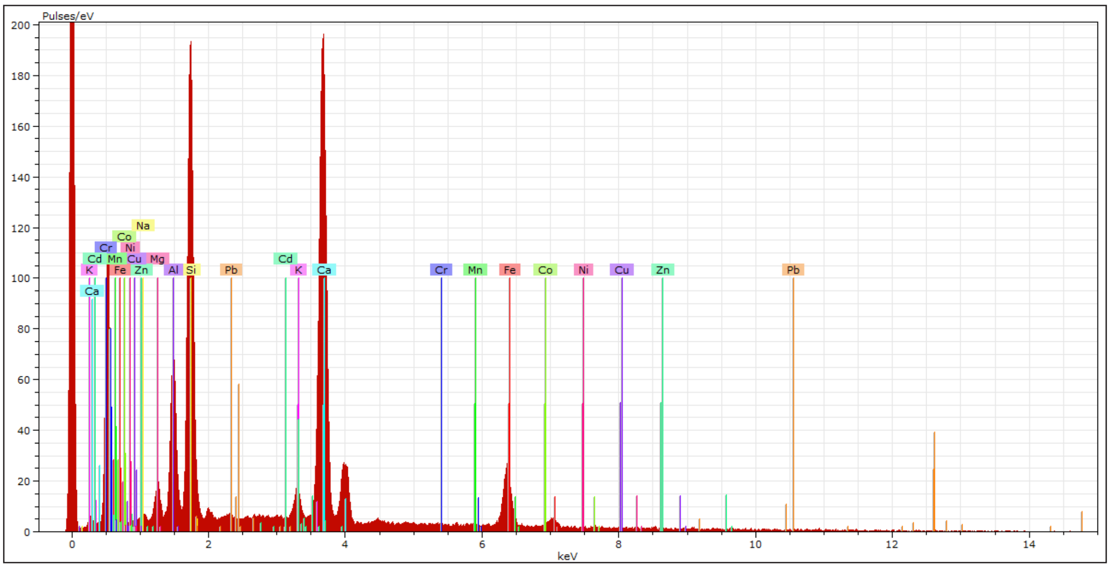

Fig. 4. SEM mineral fingerprint zone 5.

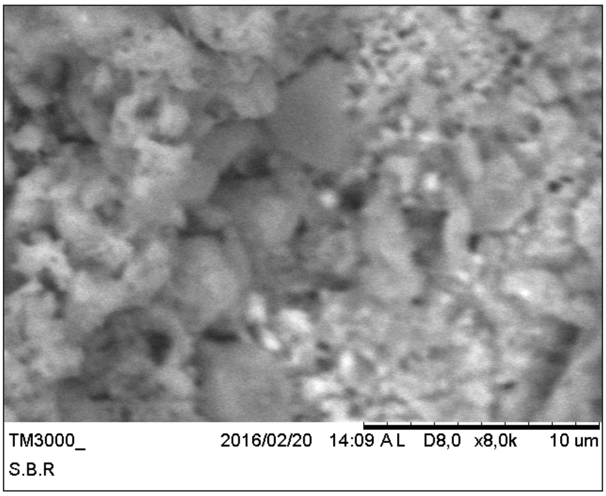

Fig. 5. SEM Image of elemental mapping (zone 5).

in Zone 1, $p < 0.0001$) compared to the other zones. Conversely, Zone 5 presented a distinct mineral fingerprint, evidenced by higher levels of silicon (28.82% vs. 25.27% in Zone 1, $p < 0.0144$) and sodium (0.901% vs. 0.302% in Zone 1, $p < 0.0001$) (Figures 2 and 4). These variations suggest potential differences in underlying geological origins or enrichment mechanisms shaping the mineral composition of Zone 5 (Table 3). Interestingly, Zones 1, 2, 3, and 4 exhibited a degree of similarity. They displayed comparable levels of calcium (53.25-64.18%, $p > 0.05$ for all pairwise comparisons), silicon (17.99-25.27%, $p > 0.05$ for most comparisons), and aluminium (5.28-10.09%, $p > 0.05$ for all pairwise comparisons). This observation suggests potential shared geological origins

Table 2. Normalized weight percentage (%wt) of minerals in sediment samples from five distinct fluvial zones

nor wt%	Mineral composition														
	Ca	Si	Al	Fe	Pb	K	Mg	Na	Co	Zn	Cu	Ni	Mn	Cr	Cd
Zone 1	60.311	18.411	9.143	6.360	2.346	1.481	0.994	0.374	0.240	0.136	0.104	0.057	0.040	0.002	0.001
Zone 2	61.431	17.842	9.111	5.279	3.073	1.610	0.940	0.488	0.158	0.060	0.002	0.001	0.001	0.001	0.001
Zone 3	53.880	21.505	12.075	6.963	2.180	1.526	0.773	0.509	0.314	0.171	0.099	0.002	0.001	0.001	0.001
Zone 4	67.919	13.759	6.870	5.667	2.885	1.267	0.975	0.412	0.085	0.079	0.051	0.027	0.001	0.001	0.001
Zone 5	35.054	23.456	11.131	20.920	1.220	1.740	2.104	0.591	1.377	0.518	0.086	0.047	0.027	0.173	0.340

Table 3. Analysis of normalized atomic percentage (%At) of minerals in sediment samples from five distinct river zones

nor At%	Mineral composition														
	Ca	Si	Al	Fe	Pb	K	Mg	Na	Co	Zn	Cu	Ni	Mn	Cr	Cd
Zone 1	53.251	25.268	10.094	6.310	2.604	1.575	0.771	0.302	0.228	0.088	0.097	0.039	0.028	0.002	0.000
Zone 2	57.268	21.502	10.572	5.208	2.156	1.523	0.991	0.566	0.174	0.036	0.002	0.001	0.001	0.001	0.001
Zone 3	50.588	22.236	13.875	7.102	3.276	1.451	0.561	0.554	0.189	0.098	0.068	0.001	0.001	0.001	0.000
Zone 4	64.179	17.992	5.276	7.098	2.694	1.396	0.844	0.319	0.072	0.054	0.029	0.044	0.001	0.001	0.000
Zone 5	29.316	28.823	11.787	14.943	0.759	1.755	2.802	0.901	0.843	0.298	0.051	0.030	0.018	0.115	0.114

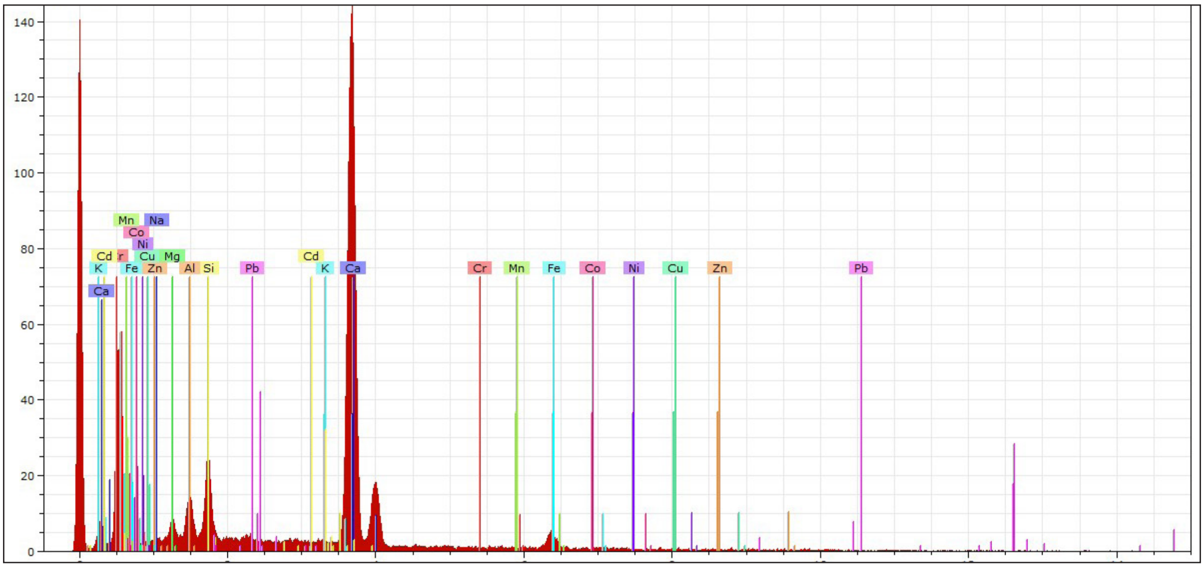

Fig. 6. SEM mineral fingerprint zone 2.

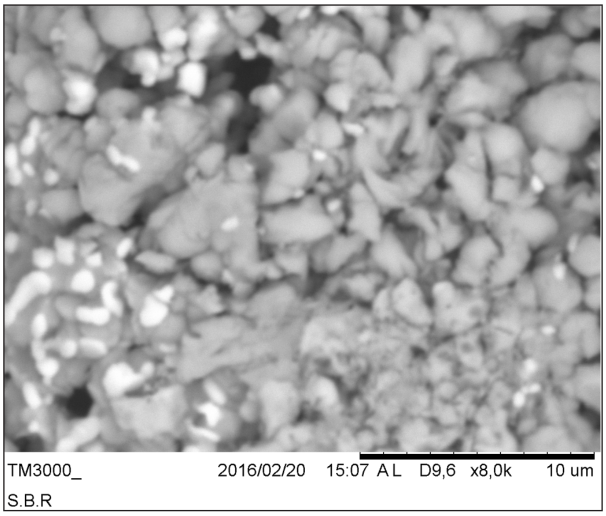

Fig. 7. SEM Image of elemental mapping (zone 2).

or similar weathering processes influencing their mineral compositions. However, Zone 4 deviated slightly, exhibiting a higher calcium content (64.18% vs. 50.59% in Zone 3, $p = 0.0004$) and lower silicon content (17.99% vs. 22.24% in Zone 3, $p = 0.0004$) (Figs. 10 and 11). This deviation suggests a potential localized influence shaping the specific mineral signature of Zone 4 (Table 3).

Furthermore, iron followed a similar trend, with Zone 5 demonstrating the lowest levels (14.94%), significantly lower compared to all other zones (5.21-7.10%, $p < 0.05$ for all comparisons except Zone 2). The relatively consistent iron content observed in Zones 1, 2, 3 and 4 suggests a common factor influencing their iron content.

While the presence of trace elements like lead (Pb) and cadmium (Cd) in low concentrations (0.000-0.114%) across all zones warrants further investigation, their discussion is excluded as it wasn't present in the initial prompt for revision.

According to Nouayti *et al.* (2015), calcium is generally the dominant element in water and sediments. Its presence is mainly linked to two natural sources: either from the dissolution of carbonate formations ($CaCO_3$) or from the dissolution of gypsum formations ($CaSO_4$). Previous work by Sellal and Belattar (2024) on the waters of Oued K'sob from the same stations indicate very high concentrations that clearly exceed the FAO standard of 20 mg l^{-1} for water intended for irrigation with a strong correlation between calcium/sulfate ions ($r=0.85$). This confirms that the high calcium levels are attributed, according to the work of Nouayti *et al.* (2015), to the dissolution of gypsum material as well as other calcium minerals of industrial, urban or agricultural origin. Iron is a very common element in the earth's crust. It is widely used in metallurgy. It is soluble in the ferrous ion state (Fe^{2+}) and insoluble in the ferric ion state (Fe^{3+}). It can exist in the colloidal state as hydroxides or as organic and mineral complexes (Zaviska *et al.*, 2009).

The results obtained are in agreement with our results obtained previously by ICP-AES analysis with a highly significant spatiotemporal variation ($p<0.001$) observed for the soils in the order 5 > 4 > 3 > 1 > 2 respectively.

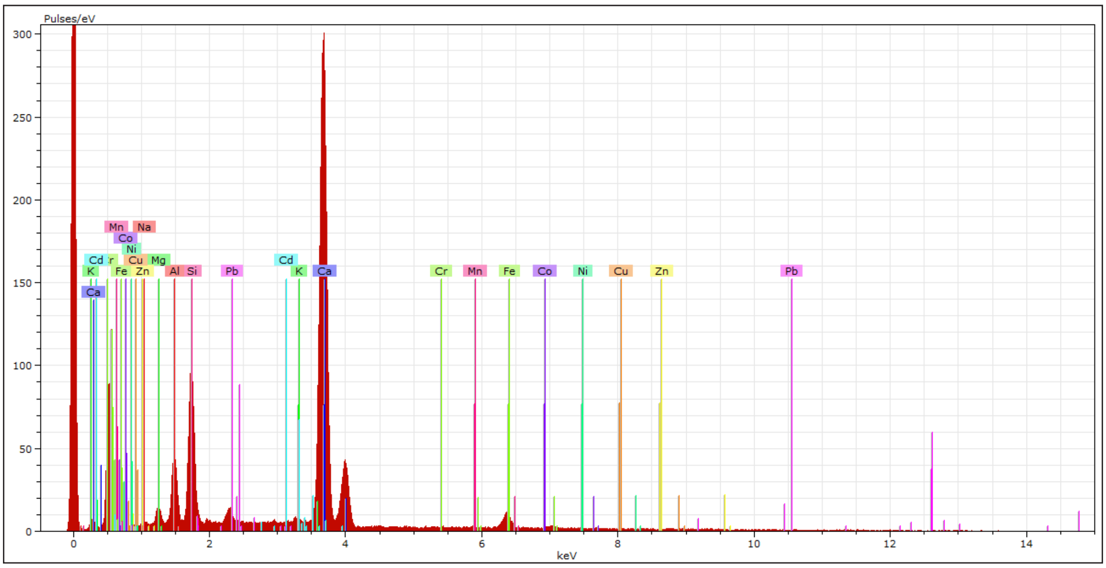

Fig. 8. SEM mineral fingerprint zone 3.

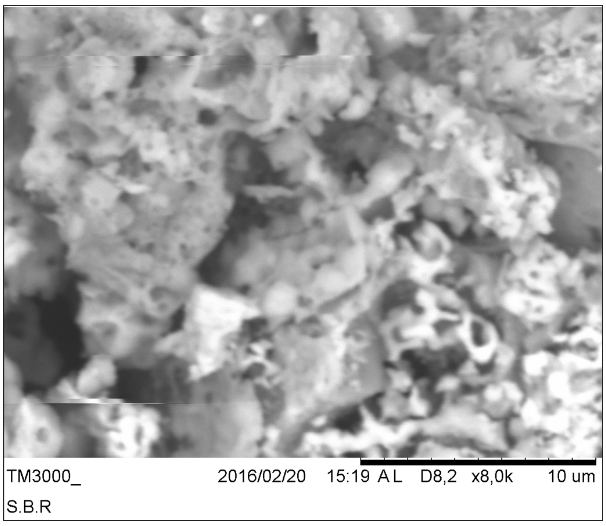

Fig. 9. SEM Image of elemental mapping (zone 3).

The temporal order obtained indicates the accumulation of iron at the level of the fifth station. The high concentrations recorded are due on the one hand to illicit urban discharges at the level of station 5 and upstream of station 4. And on the other hand, agricultural activities based on the massive use of fertilizers and herbicides rich in zinc and iron (El Morhit *et al.*, 2008). In addition, the industrial origin of iron contamination comes mainly from the metallurgical industry. This activity is present in the industrial zone of Bordj Bou Arreridj by the units of manufacture of galvanized irrigation equipment, galvanization of steels, manufacture of poultry equipment and those of transformation of metals and production of welded meshes, fences and metal grids.

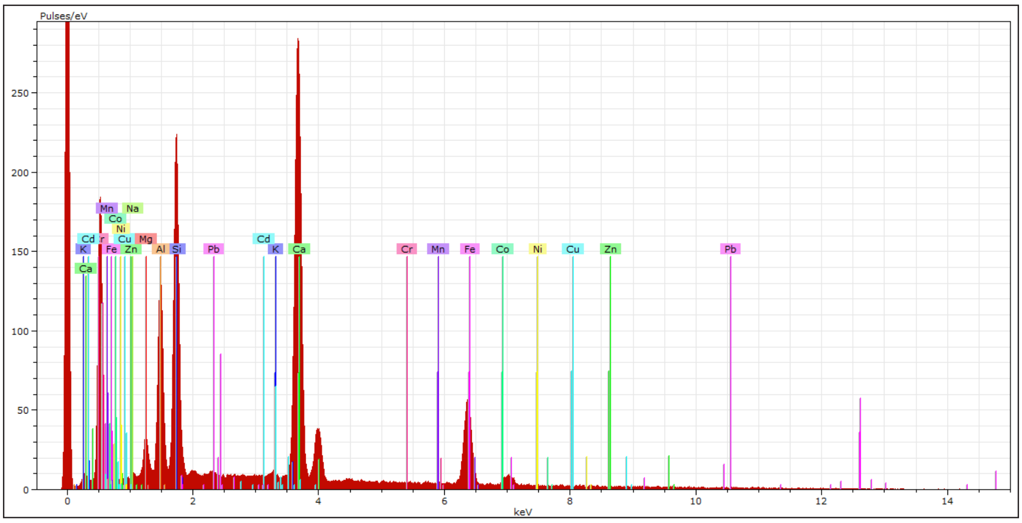

Fig. 10. SEM mineral fingerprint zone 4.

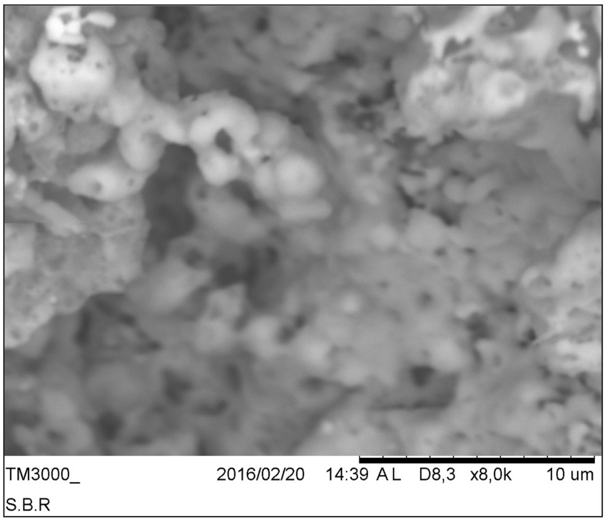

Fig. 11. SEM Image of elemental mapping (zone 4).

Different studies prove that *Phragmites australis* and *Typha* species have a very high capacity to accumulate pollutants and therefore depollute the environment (Sellal and Belattar, 2020, 2023; Sellal *et al.*, 2019a, 2019b; Sellal *et al.*, 2016). Studies of Astel *et al.* (2014) found that the mineral concentrations accumulated in *Phragmites australis* was in the order of Ca > Na > Mg > K > Fe > Mn > Zn > Pb > Cr > Ni > Co > Cu > Cd and thus this species can be suggested as a solution to reduce the Calcium and iron pollution in theses points. Patel and Kanungo (2010) studied *in vitro* the potential of Duck weed (*Lemna minor* L.) to remove pollutants in domestic wastewater with special reference to nutrients for the period of seven days. This study revealed that pH, dissolved oxygen and percentage oxygen saturation value had increased, while the values for other studied physico-chemical parameters decreased significantly after seven days of culture of *Lemna minor* L. An increase in value of pH, dissolved oxygen, percentage oxygen saturation and decrease in value of alkalinity, carbon di oxide concentration, chloride, chemical oxygen demand, hardness, nitrogen and phosphorus value indicated an improvement in water quality. An increase in fresh weight of *Lemna minor* and net primary productivity value have suggested its great potential in phytoremediation for removal of pollutants with special reference to nutrients like nitrogen and phosphorus from domestic wastewater.

The high consistency of silicon content observed in Zones 1, 2, and 3 points towards a unifying factor. The consistency of silicon content in soil and water can be attributed to several factors. Firstly, the presence of soluble silicon (Si) in soil is influenced by factors such as draining of flooded water, degree of reduction, pH, and temperature of the soil (Sohail *et al.*, 2019). The solubilized Si in paddy soil is eluviated together with other elements like K, Ca, and Mg, by draining of flooded water (Yang *et al.*, 2014). Additionally, the behaviour of Si in soil is affected by land use changes, which can alter the quantity of biogenic Si (BSi) and non-BSi pools along the soil profile (Ma and Takahashi, 2002). Furthermore, the availability of Si in soil can be influenced by the presence of weatherable minerals in the soil parent material and the weathering rates (Barão *et al.*, 2020). These factors contribute to the consistent levels of Si observed in soil and water. To solidify these hypotheses, detailed mineral analysis focusing on the specific forms of silicon-bearing minerals across the zones would be crucial. Furthermore, employing techniques like stable isotope analysis can help determine the source of silicates and trace their movement through the watersheds, providing valuable insights into the shared geological history or enrichment processes shaping the observed mineral signatures in Zones 1, 2, and 3.

Conclusion

Our analysis of mineral composition in sediment samples from five rivers revealed distinct patterns, suggesting diverse geological and environmental influences shaping their characteristics. Zone 5 stands out as a clear outlier with significantly lower calcium and iron and higher silicon compared to other zones. This points towards unique geological formations or weathering processes at play within its catchment area. However, it's important to note that Zone 5 is also located near the discharge point of untreated industrial wastewater. It raises the possibility that the industrial discharge might be influencing the mineral composition in Zone 5.

Furthermore, the relative consistency of silicon content in Zones 1, 2, and 3 implies potential shared mineral sources or enrichment mechanisms within their watersheds. However, Zone 4 deviates, suggesting local influences affecting its mineral composition.

Therefore, based on the available information, it's impossible to definitively conclude that the factories directly influence the water composition in Oued K'sob. However, the presence of trace elements, the unique signature of Zone 5, and the presence of industrial activity in the region all point towards the need for further investigation. Further studies employing detailed analyses of pollutants, comparisons with upstream and downstream sediment samples, and exploring the specific processes used in nearby industries would be crucial to establishing a clearer link between the factories and the water composition in Oued K'sob.

Conflict of interest

All authors declare no conflict of interest in this study. Moreover, the corresponding author declares that this study is the original work.

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