Complex resistivity spectrum of pollutant soils with low-concentration heavy metals

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ARTICLE INFO

ABSTRACT

Soil contamination by heavy metals occurs globally, with varying degrees of severity, especially in agricultural fields. Investigating the frequency response characteristics of different types of heavy metal pollutants through induced polarization can provide valuable evidence for surveys based on this method. Soil specimens with varying low concentrations of copper (Cu), chromium (Cr), cadmium (Cd), and lead (Pb) heavy metals were prepared for this study, and parameters including complex resistivity, amplitude-frequency, and resistivity phase were measured. Our findings reveal the following trends: Complex resistivity decreases as heavy metal concentrations increase, demonstrating significant shifts within lower concentration ranges but presenting limitations for assessing pollution in high-concentration areas. Conversely, amplitude-frequency increases with higher heavy metal concentrations, displaying excellent performance in high-concentration scenarios. The differences in complex resistivity and amplitude-frequency among different types of heavy metal pollutants are distinct. In contrast, the absolute phase decreases with rising heavy metal concentrations. The resistivity phase spectra for various heavy metal pollutants exhibit unique patterns. For example, copper-contaminated soil exhibits phase peaks in the frequency range of 8–32 Hz, whereas chromium-contaminated soil shows phase peaks at 16–64 Hz. Cadmium-contaminated soil displays phase peaks ranging from 0.25 Hz to 2 Hz, while lead-contaminated soil exhibits phase peaks within the 0.5 Hz–4 Hz range. Leveraging the frequency range corresponding to phase peaks as an identification method for heavy metal pollution types proves effective. The frequency response characteristics of induced polarization vary significantly among different types and concentrations of heavy metal pollutants, providing important foundations for the application of induced polarization method in the field of heavy metal pollution detection.

1. Introduction

The escalating severity of agricultural soil pollution resulting from human activities, including mining and smelting [1,2,3,4], urbanization [5,6,7], fertilizer application [8], and domestic waste discharge, poses a substantial threat to economic and social

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sustainability. Consequently, the detection, early warning, remediation, and prevention of heavy metals in soil have emerged as critical environmental concerns that demand immediate attention from the general public, government, and academia [9,10].

Traditional techniques for detecting soil heavy metal pollution are known for their high accuracy, strong reproducibility, and established reliability. However, these methods involve the use of drilling for sampling and depend on large-scale instruments. This leads to complex procedures, high expenses, limited real-time capabilities, and a potential risk of secondary pollution [11,12,13,14].

Soil, as a porous medium, is permeated by water and other pollutants present on the surface of solid particles and within pore structures. Heavy metals are transported through the soil via water flow, thereby influencing the soil's electrical conductivity, magnetic permeability, dielectric properties, and polarization characteristics [15]. Geophysical methods hold significant potential for monitoring soil heavy metal pollution and have undergone extensive exploration.

Ground-penetrating radar (GPR) is utilized to detect the spatial distribution of heavy metal contaminants in soil by discerning variations in dielectric constants [16]. This method boasts high efficiency and resolution. However, effectively isolating GPR signals associated with pollution remains a challenge due to geological and hydrological factors. Electrical resistivity methods delve into the migration patterns and diffusion extents of heavy metal pollutants, relying on differences in conductivity [17,18,19,20]. This technique is both efficient and cost-effective. Nonetheless, it is subject to various influencing factors, with moisture content being particularly notable. In areas with low resistivity, as seen in heavy metal contamination zones, this method may result in blurred indications. Magnetic methods are employed to assess the degree and spatial extent of pollution, capitalizing on disparities in magnetism [21,22]. This approach is known for its efficiency and high sensitivity. However, magnetic field signals within polluted areas can be feeble and susceptible to interference from environmental electromagnetic sources. The selection of observation equipment and methodologies can introduce complexities in interpreting magnetic field anomalies.

The induced polarization (IP) method, is one of the most widely applied electrical exploration techniques in the field of geophysics. It has achieved considerable success in geological prospecting for minerals and groundwater detection [23]. Importantly, it is not significantly affected by variations in topography or the uneven distribution of rock conductivity. The IP method possesses the ability to discriminate between chargeable anomalies caused by electronic conductors and those induced by ionic conductors. Given these attributes, it holds significant potential for indicating heavy metal pollution. However, its application in the field of heavy metal pollution detection has been relatively scarce in the existing literature.

In recent years, preliminary attempts have been made to apply IP in the monitoring of heavy metals in soil, accumulating valuable experience in this field. Lorne et al. derived from a double-layer model that the frequency corresponding to the phase peak is proportional to the ion diffusion rate and independent of the conductivity of the pore water [24]. Ntarlagiannis investigated the relationship between mineralization processes and spectral-induced polarization signals, exploring the potential application of spectral-induced polarization in monitoring mineralization and microbial activity [25]. The project team led by Nai conducted indoor experiments and field tests on the complex resistivity of chromium-contaminated soils, making pioneering contributions to the study of frequency dispersion characteristics, mathematical inversion of models, and detection techniques for complex resistivity [26,27,28]. Li conducted an application study of the contact-induced polarization method in detecting leachate targets. By comparing physical simulation experiments in soil tanks with traditional induced polarization observation data, the study verified the efficient and high-precision capability of the contact-induced polarization method in detecting heavy metal-contaminated soils [29].

Soil pollution presents a multifaceted challenge, and acquiring a thorough and precise comprehension of it relies on the adoption of innovative monitoring techniques. The conventional time-domain induced polarization (IP) method entails the measurement of secondary potentials produced by the IP effect following the discontinuation of the excitation current. To capture measurable secondary potentials, a heightened excitation current is necessitated, resulting in inherent drawbacks such as elevated power consumption and cumbersome equipment. In contrast, the frequency-domain induced polarization (FDIP) method offers benefits like portability and robust resistance to interference. Nonetheless, numerous existing FDIP instruments still employ variable-frequency transmissions, requiring sequential adjustments in frequency. This sequential frequency alteration diminishes operational efficiency and engenders notable disparities in measurement accuracy across various frequencies. Conversely, the pseudo-random signal IP approach designs current waveforms encompassing multiple primary frequencies. This facilitates the acquisition of amplitude and phase spectra for numerous frequencies generated by the polarization effect using a single power supply. This methodology not only expedites IP spectra measurement but also considerably enhances the relative accuracy and interference resilience of data collected at neighboring frequencies.

Current research has primarily conducted experiments and analyses focusing on the complex resistivity properties of individual heavy metals. There is considerable importance in extending this research to encompass various types of heavy metal pollutants. Existing scholars have extensively examined and summarized a wide range of rocks and minerals, each exhibiting distinct phase spectra [30,31,32]. One of the most frequently employed techniques for distinguishing graphite from different metal minerals is based on phase peak values. Due to the variations in composition, content, and structural morphology among different heavy metals, they are theoretically expected to display evident differences in resistivity phase spectra. This study investigates the feasibility of applying this method to the field of heavy metal pollution by fabricating soil samples containing heavy metals and conducting experiments. Moreover, existing research often concentrates on simulating experiments involving high concentrations of heavy metal pollutants, which leaves a gap in addressing low-concentration heavy metal pollution, even though heavy metals can pose threats to human health at relatively low concentrations. Therefore, utilizing the pseudo-random signal induced polarization method, this paper measures the resistivity, amplitude-frequency response, and impedance phase of low-concentration Cu, Cr, Cd, and Pb-contaminated samples. It summarizes the frequency response characteristics of different heavy metal pollutants, compares the suitability of various methods, and offers a fresh perspective for the efficient assessment of soil heavy metal pollution.

2. Materials and methods

2.1. Soil specimen preparation

The process of preparing soil specimens for heavy metal pollution analysis is depicted in Fig. 1. Following air-drying, grinding, sieving, and homogenization, the soil undergoes sampling using the quartering method. Subsequently, the soil is uniformly mixed with heavy metal reagents and carefully layered into acrylic square containers measuring 70 mm * 70 mm * 70 mm, ensuring adequate compaction.

To ensure data accuracy, the following measures are implemented during specimen preparation:

1. The weighing error for both soil and solution maintained below 0.1 g.
2. The soil and solution are meticulously mixed through uniform stirring, added incrementally.
3. Layer-by-layer stacking and compaction are carried out to ensure optimal interlayer contact.
4. Appropriate protective measures are taken to prevent inhalation or skin contact.
5. The specimen surface is kept smooth to minimize measurement errors.

2.2. Measurement instruments and experimental apparatus

The measurement instruments utilized in the study were the DGN-16 multifunction signal generator and the WSJ-4 pseudorandom signal induced polarization instrument. The transmitter and receiver were synchronized through GPS and operated at a supply voltage of 5 V. The frequency range employed spanned from 0.0625 to 4 Hz, encompassing a total of 7 frequencies with logarithmically spaced intervals ($2^{-4}\sim2^{2}$).

The experimental setup, illustrated in Fig. 2, comprised a symmetric quadrupole array. The separation distance between electrodes AB was 60 mm, while electrodes MN were spaced at 10 mm apart. The power supply was connected to plate-shaped copper electrodes (dimensions: 30 mm*40 mm), whereas rod-shaped copper electrodes (diameter: 2 mm) were employed to measure the potential difference.

2.3. Experimental content

This study establishes seven concentrations for Cu, Cr, Cd, and Pb, with a concentration of 0 mg/kg serving as the background level (Table 1).

The soil was weighed (430 g), and the necessary amounts of heavy metal ion solution and distilled water for each specimen were calculated based on the concentration of heavy metals and volumetric moisture content. After thorough mixing and stirring, the specimens were prepared for testing, sealed, and left undisturbed for 48 h prior to measurement. For each of the four heavy metals, we utilized seven concentrations and prepared three specimens for each concentration, resulting in a total of 84 specimens of heavy metal pollutants. The measurement result was determined by taking the average value of the three specimens with the same metal and concentration.
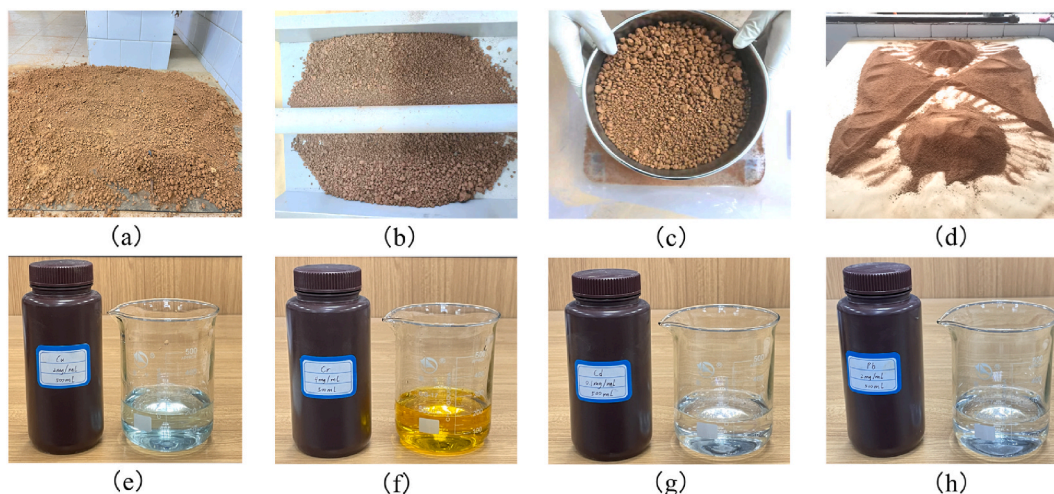

Fig. 1. (a–h). The collected soil needs to be air-dried, screened, sieved and then sampled, and copper, chromium, cadmium, and lead are used for heavy metal reagents.
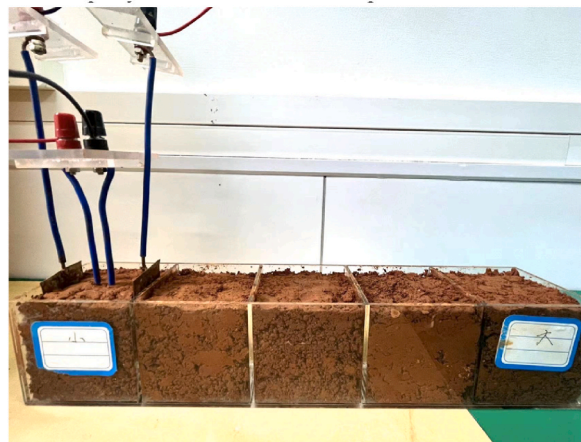
Fig. 2. The experimental setup used 2copper sheet electrodes and 2copper wire electrodes to completely insert into the soil.

Table 1
Specimen parameter settings.

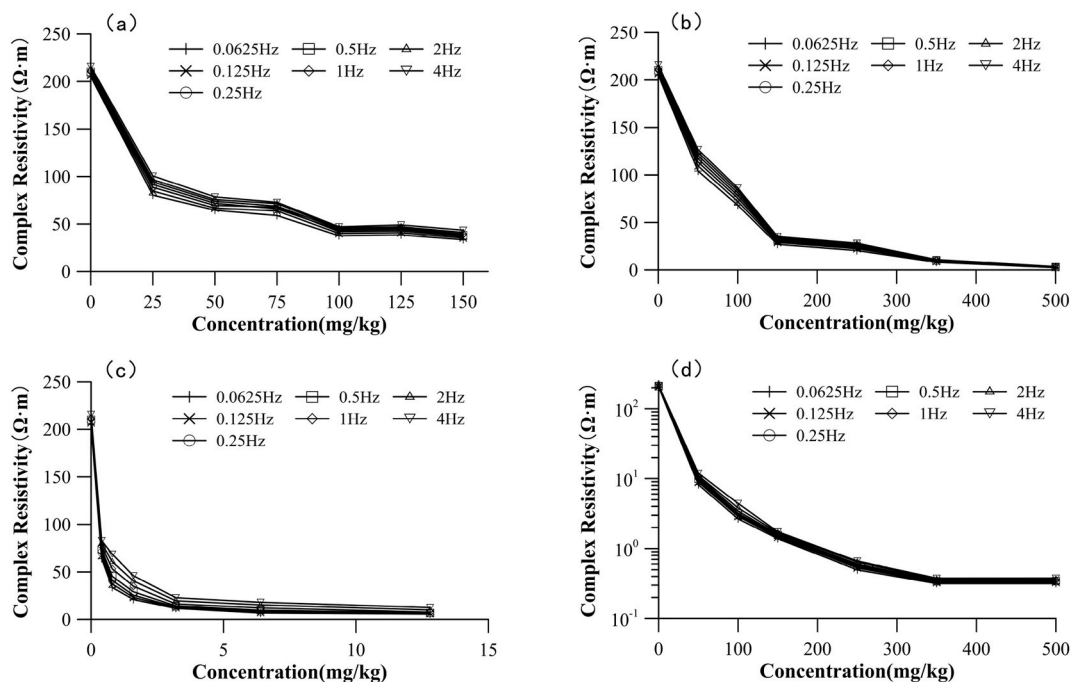| Heavy metal	Concentration (mg/kg)						
Cu	0	25	50	75	100	125	150
Cr	0	50	100	150	250	350	500
Cd	0	0.4	0.8	1.6	3.2	6.4	12.8
Pb	0	50	100	150	250	350	500

Fig. 3. Concentration-complex resistivity curves of soil specimens containing Cu (a) , Cr (b) , Cd (c) and Pb (d) . The curves shows that an increase in the concentration of all heavy metals reduce resistivity, especially Cd and Pb. The complex resistivity at each frequency reflects the same information.

3. Results and discussion

3.1. Complex resistivity

At a concentration of 0 mg/kg, the specimens exhibit a complex resistivity ranging from 205.04 to 215.88 Ω m. As the concentration of heavy metals increases, there is a sharp decrease in complex resistivity. Specifically, for a copper (Cu) concentration of 50 mg/kg, the complex resistivity ranges from 64.54 to 78.54 Ω m (Fig. 3a). Similarly, for a chromium (Cr) concentration of 50 mg/kg, the complex resistivity ranges from 104.11 to 126.71 Ω m (Fig. 3b). For cadmium (Cd) concentration of 0.4 mg/kg, the complex resistivity ranges from 63.82 to 82.98 Ω m (Fig. 3c). Lastly, for a lead (Pb) concentration of 50 mg/kg, the complex resistivity ranges from 8.18 to 11.95 Ω m (Fig. 3d). These changes in complex resistivity can be attributed to the increased conductivity of the pore water resulting from the presence of heavy metal ions.

From a comprehensive standpoint, notable disparities in the complex resistivity of soil specimens contaminated with the four heavy metals are observed. Utilizing complex resistivity as a means to differentiate between mild heavy metal contamination types and evaluate the risk standards of heavy metal pollution proves to be a viable approach. However, beyond a certain threshold concentration, the complex resistivity no longer undergoes substantial changes. Complex resistivity is primarily suitable for qualitative indication of pollution presence, as multiple factors in real-world field conditions can give rise to variations in resistivity. The moisture content stands out as the most influential factor, thereby rendering resistivity measurements ineffective in scenarios such as riverbanks and irrigated paddy fields.

3.2. Amplitude-frequency

Amplitude-frequency, commonly referred to as the polarization rate, is a term frequently employed in this field. Conventional time-domain induced polarization (IP) methods assess soil pollution by calculating the polarization rate at a single frequency based on potential differences observed at various time intervals. Nevertheless, this approach is vulnerable to interference. In our study, we compute the polarization rate by concurrently analyzing potential differences generated at two different frequencies. This method enhances accuracy and resistance to interference. Specifically, this paper examines amplitude-frequency at three frequency pairs: Fs17 (0.0625 Hz and 4 Hz), Fs14 (0.0625 Hz and 0.5 Hz), and Fs47 (0.5 Hz and 4 Hz).

As depicted in Fig. 4a-d, when the concentration of heavy metals is 0 mg/kg, the specimens display an amplitude-frequency response ranging from 0.13% to 0.37%, indicating weak polarization effects. As the concentration of heavy metals increases, the polarization effects of the specimens intensify, leading to higher amplitude-frequency values. The frequency pairs Fs17, Fs14, and Fs47 all exhibit an upward trend in amplitude-frequency response with increasing concentration. Notably, different types of heavy metals show significant variations. In the case of Cu and Cr soil specimens, the order of amplitude-frequency values is Fs17 > Fs14 > Fs47. The greater the frequency difference, the higher the amplitude-frequency, indicating a more complete occurrence of the induced polarization effect. Larger frequency differences correspond to higher amplitude-frequency values. Moreover, for a given frequency difference, lower frequencies correspond to higher amplitude-frequency values, indicating a more pronounced polarization effect of Cu and Cr pollutants at lower frequencies. As for Cd and Pb, the amplitudes of Fs14 and Fs47 are quite similar, but overall, Fs17 demonstrates the highest amplitude value.
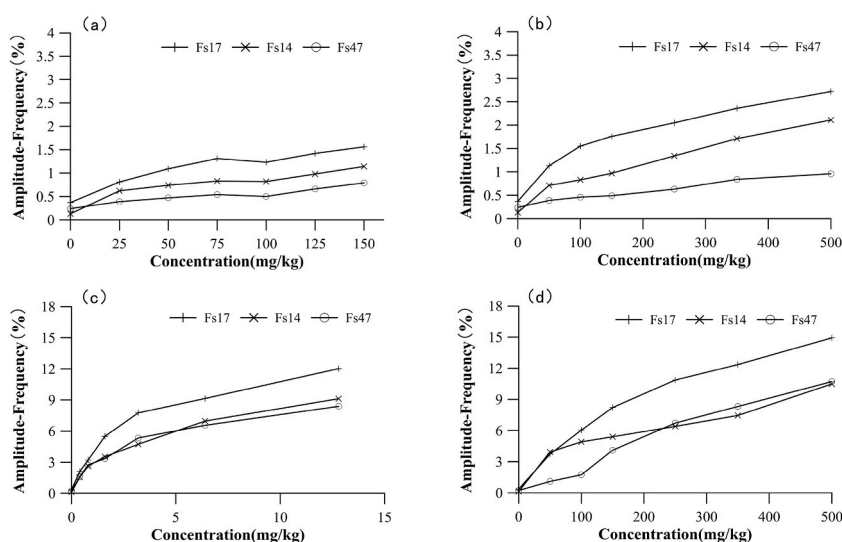

Fig. 4. Concentration-amplitude-frequency curves of soil specimens containing Cu (a) , Cr (b) , Cd (c) and Pb (d) . The curves shows that an increase in the concentration of all heavy metals increase amplitude-frequency, especially Cd and Pb. A broader frequency range unveils distinct induced polarization responses to heavy metal pollution.

When investigating soil heavy metal pollution using pseudo-random signal induced polarization method, it is recommended to utilize the amplitude-frequency response with larger frequency differences as the standard. This parameter provides more accurate reflection of the soil's polarization phenomenon. Examining the amplitude values of the amplitude-frequency response (taking Fs17 as an example), the amplitude-frequency response of Cu soil specimens ranges from 0.81% to 1.56% (concentration range: 25 mg/kg to 150 mg/kg). For Cr soil specimens, it ranges from 1.13% to 2.72% (concentration range: 50 mg/kg to 500 mg/kg). Cd soil specimens exhibit a range of 2.12%–12.02% (concentration range: 0.4 mg/kg to 12.8 mg/kg), while Pb soil specimens show a range of 3.73%–14.93% (concentration range: 50 mg/kg to 500 mg/kg). The amplitude-frequency of Cd and Pb displays significant variations in response to changes in heavy metal concentration. Additional experimental research holds the potential to establish a quantitative correlation between the amplitude-frequency and heavy metal concentration. This could facilitate the creation of standard concentration values, thereby augmenting the applicability of this method.

3.3. Resistivity phase

3.3.1. Absolute phase

Traditional induced polarization methods primarily focus on studying the absolute phase.

According to Fig. 5a-d, when the concentration of heavy metals is 0 mg/kg, the specimen's absolute phase approaches 0, indicating a weak induced polarization effect. The absolute phase curve of Cu soil specimens exhibits a serrated pattern, suggesting the presence of nonlinear effects. Similar phenomena are observed for Cr and Pb soil specimens at a concentration of 100 mg/kg. These occurrences in the phase spectrum are attributed to the varying nonlinear effects generated at the same frequency when supplied with multi-frequency currents. The relationship between concentration and phase may represent a specific and meaningful phenomenon [33].

All four soil specimens demonstrate a trend where higher concentrations of heavy metals correspond to smaller absolute phases. This confirms that as soil contamination by heavy metals worsens, the induced polarization effect becomes stronger. Therefore, the absolute phase can be used to infer the degree of soil contamination by heavy metals. Furthermore, there are variations in the absolute phase magnitudes at different frequencies for distinct heavy metals. Specifically, at low frequencies (0.0625 Hz), Cu and Cr demonstrate the smallest phase values, while Cd and Pb exhibit the largest phase values. The interrelationships among phases at each frequency are intricate and convey valuable information, which will be elaborated upon in the subsequent phase spectra.

3.3.2. Phase spectra

In the absence of heavy metal contamination (0 mg/kg), the resistivity phase remains nearly constant near zero across the entire frequency range. With increasing concentrations of heavy metals, the phase spectra for the four metals display unique patterns and shift upward collectively. This upward shift correlates with a reduction in the absolute phase, signifying an intensified induced polarization effect. Notably, the shapes of the spectra remain unaltered despite variations in concentration. As shown in Fig. 6a-b, the
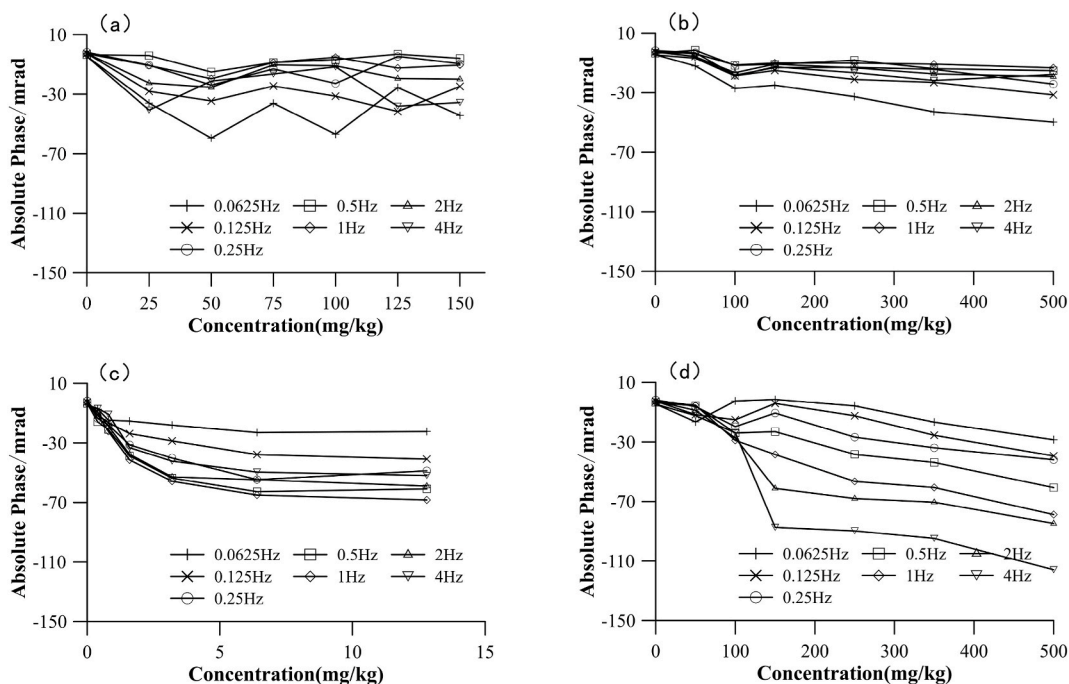

Fig. 5. Concentration-phase curves of soil specimens containing Cu (a) , Cr (b) , Cd (c) and Pb (d) . The curves shows that an increase in the concentration of all heavy metals reduce absolute phase.
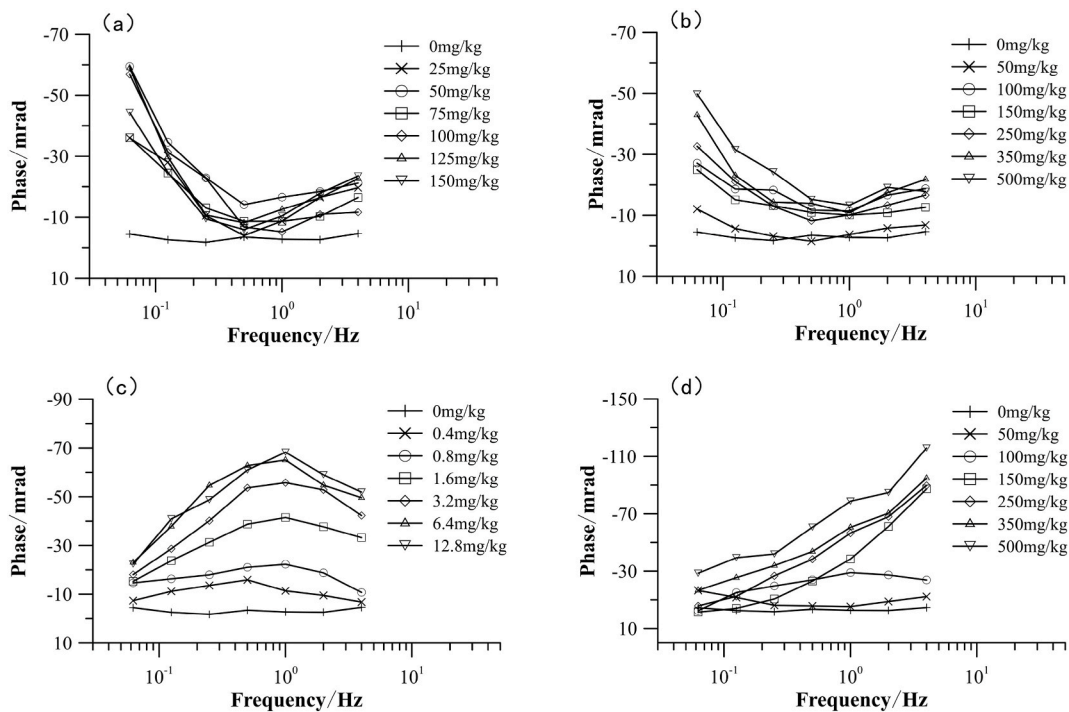
Fig. 6. Phase spectrums of soil specimens containing Cu (a) , Cr (b) , Cd (c) and Pb (d) .The curves shows that the phase spectrums of different heavy metals are different, and extreme phases occur in the different frequency ranges, which are not affected by concentration.

phase spectrum of Cu and Cr soil specimens exhibits prominent peaks ranging from 0.25 Hz to 2 Hz. Conversely, Cd soil specimens display an inverse pattern, with the lowest phase values observed in the low-to-mid frequency range, specifically showing a minimum peak within the frequency range of 0.5 Hz–4 Hz (Fig. 6c). The phase spectrum of Pb soil specimens demonstrates a different trend, appearing relatively flat at concentrations of 50–100 mg/kg (Fig. 6d). However, as the concentration exceeds 50 mg/kg, the phase values decrease with increasing frequency, with higher frequencies exhibiting lower phase values.

Comparing these findings with the phase spectra of natural specimens from various mineral species, as tested by Zhang et al. it can be inferred that the minimum values of the phase spectra of Pb soil samples would occur in a higher frequency range [34]. Subsequent experiments have confirmed this conjecture.

Distinct variations are observed in the phase spectra among different heavy metals. However, due to the limited frequency range, specific characteristic frequencies associated with the polarization effect of certain heavy metals were not identified. To address this limitation, experiments were conducted using the GSZA-FW01 Full-Waveform Impedance Analyzer. Cu, Cr, and Pb soil specimens with a concentration of 200 mg/kg, along with Cd soil specimens with a concentration of 20 mg/kg (while keeping all other conditions
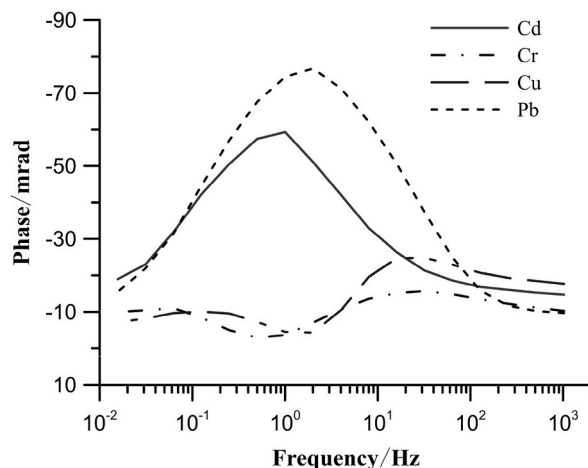

Fig. 7. Phase spectrums of soil specimens containing different Heavy metals in a wide frequency range.

constant), were examined within the frequency range of 0.01563–1024 Hz ($2^{-6} \sim 2^{10}$Hz).

Fig. 7 illustrates the peak frequencies for Cd and Pb, which occur within the ranges of 0.25–2 Hz and 0.5–4 Hz, respectively. For Cu and Cr, the peak values are observed at frequencies of 8–32 Hz and 16–64 Hz, respectively. Additionally, Cu exhibits significant values at 0.5–2 Hz, while Cr shows significant values at 0.25–1 Hz. In both the lower and higher frequency ranges, the phase values for all four heavy metals tend to converge towards zero. These findings highlight distinct differences in the phase spectra among the various heavy metals. Consequently, identifying the types of heavy metal pollution based on the frequency range corresponding to the phase peak values proves to be an effective and meaningful approach.

3.4. Discussion

Soil-induced polarization characteristics are effective indicators of heavy metal pollution. As heavy metal concentrations rise, soil resistivity decreases, apparent frequency increases, and absolute phase decreases. Extensive experiments have the potential to establish explanatory models linking these parameters to varying heavy metal concentrations. It's worth noting that as heavy metal concentrations increase beyond certain levels, the trend in resistivity change becomes less pronounced. In some cases, especially in areas with higher moisture content, this can lead to a low-resistivity anomaly that blurs signals of heavy metal contamination. Additionally, at lower concentrations, apparent frequency tends to have a lower amplitude and may not precisely reflect pollution levels. However, when these two parameters are combined, they complement each other. Resistivity is more effective at indicating low-concentration pollution, while apparent frequency is better suited for high-concentration pollution. Experimental results demonstrate that apparent frequency continues to display significant variations at higher concentrations, with minimal sensitivity to water content. This establishes apparent frequency as a reliable indicator of heavy metal pollution in riverbanks and irrigated farmlands.

In the process of preparing soil pollution samples, it's crucial to maintain an appropriate moisture level and ensure the even distribution of heavy metals within the soil. Because laboratory analyses involve small soil samples, and any irregularities or non-uniformities in these samples can be amplified, potentially impacting the experimental results. However, when conducting tests on natural soils in field settings, these constraints are not applicable. Fieldwork operates on a larger scale, typically around 100 m * 100 m or larger. The primary objective is to keep the ground resistance low at the power supply electrode and achieve a consistent and stable current distribution across the entire work area. If the field soil is dry, a solution of saltwater can be applied at the power supply and measurement electrode locations to enhance the contact between the electrodes and the soil, consequently reducing ground resistance. The two measurement electrodes capture induced polarization data from the entire soil area between them. This approach ensures consistent operation, even in regions between the electrodes where variations in scale and soil moisture content may exist, all while maintaining the accuracy of the work.

Experimental data supports the notion that, similar to rocks and minerals, various heavy metals display distinctive characteristics in their phase spectra. Employing the frequency range associated with phase peak values to identify types of heavy metal pollution has been validated as an effective and meaningful method. However, current pseudo-random signal systems can only simultaneously generate seven frequencies, resulting in a relatively narrow frequency band. Consequently, the characteristic polarization frequencies of some heavy metals remain undetected. On the other hand, although other spectral induced polarization instruments can function across a wider frequency spectrum, they rely on variable-frequency transmissions, significantly extending their operational duration to over ten times that of pseudo-random signal induced polarization instruments. Hence, the augmentation of emission frequencies in pseudo-random signal systems holds paramount significance for the expeditious and highly precise assessment of soil heavy metal pollution.

4. Conclusion

(1) Various heavy metals display significant differences in their electrical conductivity. Resistivity experiences noticeable fluctuations within low concentration ranges. Nonetheless, relying solely on resistivity for assessing heavy metal-related pollution, especially in regions with high moisture content, has its limitations. To overcome these constraints, it can be beneficially complemented with amplitude-frequency measurements derived from the induced polarization method, as amplitude-frequency exhibits a strong sensitivity to high-concentration heavy metal pollution.

(2) Across a broad frequency spectrum, various heavy metals manifest distinctive phase spectra, displaying stark deviations from spectra of uncontaminated soil. Alterations in concentration minimally affect spectral morphology, with the frequency range linked to phase peak values demonstrating relative stability. Employing this frequency range associated with phase peaks proves efficacious and meaningful for discerning types of heavy metal pollution. Furthermore, the magnitude of phase spectra, assessed through absolute phase values, can offer insights into the extent of heavy metal contamination, albeit to a limited extent.

(3) In contrast to the conventional spectral induced polarization method, the pseudo-random signal induced polarization method substantially improves observational accuracy and operational efficiency through the simultaneous emission of multiple frequency signals. Nevertheless, its frequency range is somewhat limited, which restricts its capacity to discern the distinctive polarization frequencies of a wider array of heavy metals. Consequently, elevating the emission frequencies of the pseudo-random signal system holds utmost significance for expeditious and highly precise soil heavy metal pollution surveys.

(4) In summary, studying the frequency effects of heavy metal pollutants' induced polarization holds great potential for accurately identifying the degree and types of heavy metal pollution in soil.

Data availability statement

Supplementary data associated with this article can be found in the online version at https://doi.org/10.17632/kn4j99334b.1.

CRediT authorship contribution statement

Xuesong Xie: Data curation, Formal analysis, Investigation, Methodology, Writing – original draft. **Diquan Li:** Formal analysis, Methodology, Project administration, Resources, Writing – review & editing. **Jiabin Yan:** Data curation, Formal analysis, Methodology, Writing – review & editing. **Jing Pei:** Data curation, Formal analysis, Project administration. **Weitian Wu:** Data curation, Investigation. **Zhongyuan Liu:** Data curation, Formal analysis, Writing – review & editing. **Xiaolin Ding:** Data curation, Writing – original draft.

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.

Acknowledgements

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