

Characterisation of metal particles in porous sand using spectral induced polarisation

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Abstract. The growing global demand of rare earth materials and efficient metal recovery techniques from natural resources highlight the significance of advanced methods for identifying and characterising metallic mineral in complex porous matrix. The study aims to characterise metal particles captured in a porous sand matrix using Spectral Induced Polarisation (SIP) method, mainly focusing on the sensitivity of SIP to variations in metal content. An experimental setup was designed to perform SIP measurements on pyrite-sand mixtures, specifically aimed at capturing the complex conductivity of the samples. This setup incorporated a custom-designed cup equipped with four embedded electrodes, facilitating four-point impedance measurements essential for analysing complex conductivity responses. Experiments were performed on sand, mixed with 3 distinct pyrite concentrations. All the measurements were performed three times to comment on the measurement variability. The SIP responses were found to vary with the concentration of pyrite particles. The results suggest that SIP is a promising method for identifying and measuring metal inclusions in porous geological materials.

1 Introduction

Rare earth elements and efficient metals are essential to meet modern requirements which includes technological applications, electronics, and advanced manufacturing. However, their occurrence within complex porous matrix poses significant challenges for efficient identification and characterisation. There is an urgent need to develop quantitative method to infer metal composition, zonation and volume at the field scale. Geophysical methods and in particular low frequency electric method such as Spectral Induced Polarisation (SIP) has the potential to fill this gap [1]. SIP is widely acknowledged as an effective geophysical method for examining the electrical characteristics of mineral bearing-porous materials. It has been used extensively in the characterisation of conductive and semi-conductive minerals, especially within complex matrices where direct chemical analysis is difficult [2]. The method is sensitive to metallic phases, allowing for the distinction of mineralogical components corresponding to their polarisation characteristics and electrical interactions with pore fluids. The SIP response is influenced by several factors which includes grain size, mineral content, and surface conductivity [3]. Previous studies have demonstrated that variations in these parameters can significantly affect phase shift, chargeability, and relaxation times, providing valuable insights into the microstructural proper-

ties of conductive mineral mixtures [4]. Previous research has concentrated on either single-phase mineral systems or controlled mixtures with fixed grain size distributions leaving a knowledge gap regarding how changes in mineral composition affect phase responses in heterogeneous matrices.

The use of SIP method for quantitative approach requires the development of theoretical approach to transfer the field measurements into quantitative parameters. Laboratory measurements are generally used to build such framework. This study uses experimental SIP measurement to examine the polarisation response of sand-pyrite mixtures with different pyrite concentrations to evaluate the impact of electrical phases signals. By examining the frequency-dependent phase shifts in various mixtures, this study aims to improve the identification and characterisation of rare earth minerals present in complex porous environments. The presented preliminary findings of this study enhance our knowledge of the use of SIP measurements in mineral characterisation by highlighting its potential as a non-invasive rapid technique for identifying conductive phases. By integrating laboratory experiments with theoretical modelling, future studies can refine SIP-based quantitative approaches, improving the reliability of mineral identification in natural settings.

2 Materials and methods

2.1 Materials

In this study, sand as a non-conducting porous material and pyrite as a metallic conducting material was used.

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Pyrite used in this study were sand size euhedral crystals with 98% purity, sourced from Peru through a commercial provider, whereas locally sourced fine grained sand was utilised. The grain size of pyrite was selected in the range of 212 μm to 300 μm . The grain size of sand was in the range of 150 μm to 300 μm . The selected particle size ranges for sand and pyrite were chosen to minimise segregation, and maintain consistency across samples. These sizes are typical of granular materials found in natural and engineered porous systems. The narrow size range reduces variability in packing and porosity. Additionally, provides an optimal balance between signal strength and measurement stability as more finer particles (e.g., < 150 μm) would significantly increase the surface conductivity and complexity due to clay-like behaviour, while coarser particles (> 300 μm) might reduce the polarisation signal strength due to decreased interfacial surface area. The specific gravity of sand and pyrite were 2.66 g/cm^3 and 4.94 g/cm^3 respectively. Homogeneously mixed sand-pyrite mixtures with varying pyrite content of 2.5%, 5.0% and 7.5% by volume were prepared. To saturate the sample, NaCl solution with a conductivity of $10^{-2}\text{S}/\text{m}$ was used to ensure sufficient ionic mobility for polarisation while maintaining moderate conductivity levels, allowing the detection of interfacial polarisation effects preventing the conductive component from overshadowing the SIP signal.

2.2 SIP technique

The Spectral Induced Polarisation (SIP) technique is based on the injection of a sinusoidal electric current, $I(t)$, in the soil at a range of frequency give by:

$$I(t) = |I| \sin(\omega t), \quad (1)$$

where $|I|$ is the current magnitude and $\omega = 2\pi f$ is the angular frequency. This injected current induces a voltage response, which is also sinusoidal but exhibits a phase shift (ϕ) relative to the current:

$$U(t) = |U| \sin(\omega t + \phi). \quad (2)$$

To make it easier to describe the current and voltage in terms of linear equations, they are represented by complex numbers [5]. The complex impedance Z^* is then determined from the ratio of the measured voltage to the applied current, expressed in exponential form as

$$Z^* = \frac{U^*}{I^*} = \frac{|U|}{|I|} e^{i\phi}. \quad (3)$$

The complex impedance is converted to complex electrical conductivity σ^* by using Equation 4 which characterises the electrical properties of the medium under investigation:

$$\sigma^* = \frac{1}{K_G Z^*} = |\sigma^*| e^{-i\phi} = \sigma' + i\sigma''. \quad (4)$$

here, σ' represents the real part and σ'' denotes the imaginary part of the complex conductivity. Whereas, K_G is the geometric coefficient used to account for the effect of geometry of the placement of electrodes.

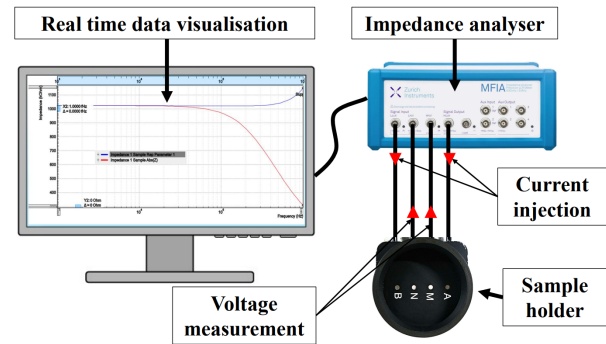


Figure 1. Experimental setup

The measured conductivity is adjusted to a standard reference temperature using the following relation:

$$\sigma(T_0) = \frac{\sigma(T)}{1 + \alpha(T - T_0)} \quad (5)$$

where $\sigma(T_0)$ is the conductivity normalised to the reference temperature T_0 (25°C), $\sigma(T)$ is the observed conductivity at temperature T , and α is the temperature correction factor, commonly taken as 0.020°C^{-1} .

2.3 Apparatus

Figure 1 shows the experimental setup used in this study. A small 3D printed cup with a height of 17 mm and diameter of 70 mm was built with 4 embedded electrodes. Non-polarising silver chloride ($\text{Ag} - \text{AgCl}_2$) electrodes were chosen to ensure precise electrical measurements. Out of the four electrodes, two were used for current injection (A and B) inside the mixture, whereas other two were utilised to measure the voltage difference (M and N). The cup is open from the top which makes the sample preparation and saturation process easier. Similar design was used by Revil et al., (2018)[6] to obtain complex conductivity of sandstone. The easier design helps to reduce the complexities that occur in the laboratory measurements. The 4-point cup is connected to the impedance analyser to measure complex impedance values of prepared mixture. To measure the complex impedance values, Multi-Frequency Impedance Analyser (MFIA) by Zurich Instruments was used in this study. The MFIA device has the ability to measure complex impedance signals from 1 m Ω to 1 T Ω , over a frequency (ω) range, from 1mHz to 500kHz. The instrument directly outputs the phase shift (ϕ) as part of the impedance spectrum acquisition. Specifically, the phase shift is defined as the argument (phase angle) of the complex impedance, measured relative to the applied voltage signal.

2.4 Sample preparation

The sand-pyrite mixtures were prepared with predetermined pyrite concentrations to ensure controlled experimental conditions. To enhance saturation and minimise

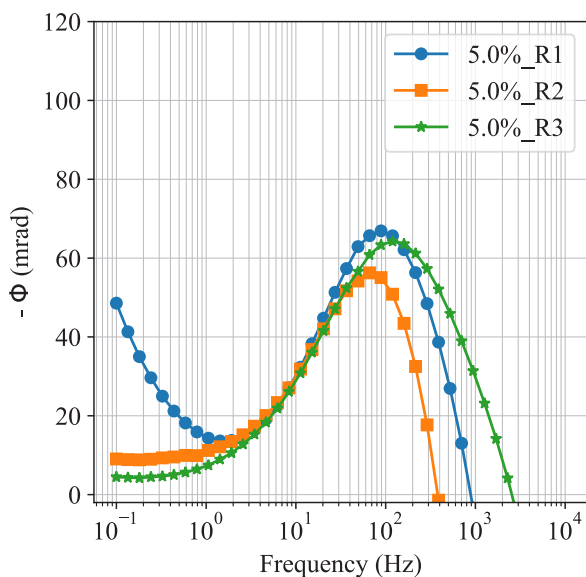


Figure 2. Phase shift as a function of frequency for 5 % pyrite content, measured over three repetitions to assess measurement variability

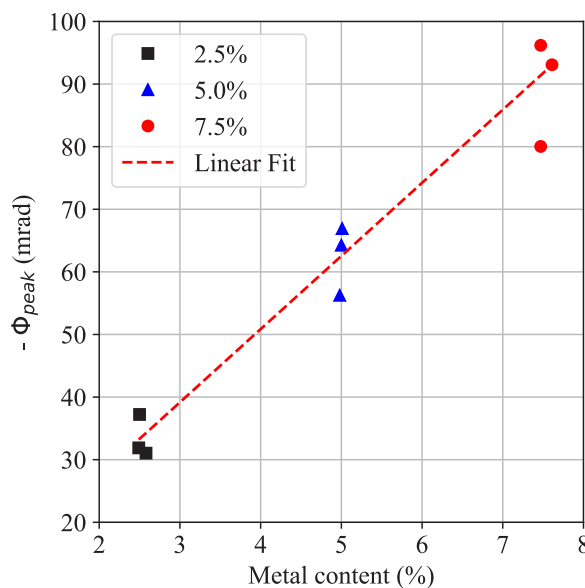


Figure 3. Obtained peak values of phase shift with varying pyrite content

air entrapment during the saturation process, the dry mixture was first blended with a conducting fluid (conductivity: 10^{-2} S/m, 5% by weight). This pre-mixing step facilitated uniform distribution of the conductive phase within the pore space. The prepared mixture was then carefully placed into the sample holder in three separate layers. Each layer was compacted using 50 uniform tamping strokes to ensure consistent density and reduce heterogeneity within the sample. The dry densities of the mixtures were measured to be approximately 1.54 g/cm^3 , 1.59 g/cm^3 , and 1.61 g/cm^3 for pyrite contents of 2.5%, 5.0%, and 7.5%, respectively. Once all layers were in place, the top surface was levelled to maintain a uniform sample height across all tests. Following compaction, the sample was gradually saturated using the same conducting fluid to prevent structural disturbance. Saturation was achieved by introducing the fluid slowly through a porous stone with the aid of a syringe, allowing capillary action to distribute the fluid evenly throughout the mixture. This method ensured that the sample structure remained intact while achieving full saturation. The saturated sample is then connected to the impedance analyser to compute the complex impedance values of the sample. 3 mixtures with varying pyrite content, 2.5%, 5.0% and 7.5% by volume were tested.

3 Results

To assess the variability of SIP measurements, all the experiments were performed three times. The repetitions were named as R1, R2, R3. Each experimental run, including all repetitions at a given pyrite concentration, was conducted using a freshly prepared batch of the sand-pyrite

mixture. No material was reused across measurements to ensure consistency and avoid any cross-contamination or alteration in material properties. Figure 2 shows the 3 repetitions of the mixture with 5% pyrite concentration. All three repetitions (R1, R2, and R3) exhibited consistent phase spectral shapes across the frequency range. Across the measured frequency range, the spectral response displayed a dispersive behaviour, indicative of interfacial polarisation processes occurring at the mineral-fluid interface. Notably, the R1 sample exhibits a secondary peak. However, this is interpreted as an anomalous measurement likely caused by sensitivity of SIP signals due to fluid distribution, boundary effects and pore structure at low frequencies [7, 8]. This behaviour lies beyond the targeted frequency range of 1 Hz to 1000 Hz, which is the focus of this study. Within this range, the observed polarisation primarily arises from interfacial charge accumulation at the mineral-fluid interface and is considered a more reliable indicator of material behaviour. The absence of this secondary peak in subsequent repetitions (R2 and R3) supports the conclusion that the deviation observed in R1 is an isolated artifact rather than a reproducible feature.

Across the three repetitions, the standard deviation in the peak phase value were 3.35 mrad, 5.54 mrad, 8.57 mrad for 2.5%, 5%, 7.5% pyrite content, respectively. This variability can be attributed to several factors, including local heterogeneity in packing, variations in pore connectivity, fluid distribution, and inconsistencies in electrode-sample contact. While a moderate level of variation is noted, it remains within acceptable bounds for preliminary laboratory studies and are sufficient to demonstrate the sensitivity of SIP phase response to changes in metal content.

The observed peak phase values across the three repetitions were found to occur within the frequency ranges of 66–290 Hz, 66–120 Hz, and 66–388 Hz for the 2.5%, 5.0%, and 7.5% pyrite mixtures, respectively. This variability in peak frequency response is primarily attributed to differences in the particle size distribution of the pyrite within each mixture. Since the polarisation frequency is influenced by the characteristic size of the conductive inclusions, non-uniformity in particle sizes leads to a broader distribution of relaxation times, thereby shifting the frequency at which maximum phase response occurs. Such variations are expected in granular systems where the metallic particles are not perfectly uniform, and they underline the importance of particle-scale properties in interpreting SIP measurements.

Figure 3 shows the obtained peak values of phase for 3 different pyrite mixtures with 2.5%, 5.0% and 7.5% pyrite concentration. Peak values of SIP responses were obtained at frequencies closer to 100 Hz for all three mixtures. The results indicate a systematic increase in the peak values of phase with increasing pyrite content. This observed trend is consistent with the theoretical expectations, as higher conductive mineral content such as pyrite enhances the polarisation effect, leading to a stronger phase shift.

4 Conclusion

This study investigated the applicability of Spectral Induced Polarisation (SIP) technique for the identification and characterisation of conductive mineral phases in sand–pyrite mixtures. Peak values of phase were observed at a frequency of approximately 100 Hz. The observed peak values increased consistently by increasing pyrite concentration. This trend aligns with expected polarisation behaviour, where higher conductive mineral content enhances charge accumulation, leading to stronger phase shifts. These results emphasise the complex connections among charge polarisation, mineral content, and frequency-dependent electrical behaviour, highlighting the necessity of more research into the mechanistic response of SIP in multi-phase systems.

To utilise SIP measurements as a quantitative tool for mineral identification, a strong theoretical foundation must be established in order to convert laboratory results into useful applications. Laboratory-based experiments, as conducted in this study, serve as a foundation for improving predictive models that relate SIP phase response to mineral composition. In addition, numerical frameworks such as Discrete Element Method (DEM) in combination with analytical electrical models (Cole-Cole models) could be used to reduce the need for extensive physical testing. The Cole–Cole model can provide a framework for quantitatively characterising the spectral shape

of the SIP response through a set of physically meaningful parameters, thereby enabling a deeper understanding of the underlying polarisation mechanisms. Future research should focus on applying the SIP technique to more complex geological environments with rare earth metal deposits.

In all, this study supports the potential of SIP as a non-invasive method for identifying and characterising conductive mineral phases in the exploration of rare earth materials. The ability to detect and differentiate mineralogical changes based on SIP phase response could support sustainable mining efforts, reducing the need for invasive sampling while improving efficiency in resource identification.

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