Monitoring of Soil Pollution in Agricultural Lands Using Magnetic Susceptibility and Mineralogy Analyses, North Al-Muthanna Province, Iraq

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Abstract
One hundred and fifty samples of soils were collected from five different agricultural lands in Al Muthanna province, southeast Iraq. This study aims to explore the magnetic mineralogy and its correlation with heavy metals in four agricultural lands in north of Al-Muthanna Province. These samples were analyzed using magnetic susceptibility and mineralogy analyses (magnetic susceptibility (χ), frequency-dependent susceptibility (κfd%), Anhysteretic remanent magnetization (χARM), S-ratio, saturation isothermal remanent magnetization (SIRM) and back field of isothermal remanent magnetization (IRM), grain size, and heavy metal analyses for soil assessment. The results show the dominance of magnetite and some portions of hematite as the main magnetic carriers in the surface soils. Super-paramagnetic (SP) and single domain (SD) grains are the two main magnetic grain sizes in the surface soils, based on cross plots of χ versus ARM and χ versus SIRM. The enhancement of magnetic susceptibility in the soil samples could be caused by pedogenic and anthropogenic processes. Heavy metal contents show no correlation with magnetic susceptibility for Rumaitha, Samawa, Warka’, while a positive correlation in Swair. This study demonstrates that magnetic mineralogy could be used as an indicator for different sources of soil contamination.

Keywords: Agricultural soils; Magnetic susceptibility; Contamination; Heavy metals; Magnetite; Al Muthanna province; Iraq

1. Introduction

Magnetic susceptibility techniques are considered non-destructive, rapid, and low-cost among all other techniques like seismic, electric and chemical methods, which can be used to assess soils. The magnetic susceptibility measurements of soils rely on three factors which are; the amount, type, and chemical composition of soil components. The magnetic susceptibility measurements for soils are sensitive to the ferromagnetic, ferrimagnetic, paramagnetic, or diamagnetic characteristics (Dearing, 1999). The magnetic minerals can be classified as: strong magnetic minerals such as magnetite (Fe₃O₄) and maghemite (γ-Fe₂O₃); Canted anti-ferromagnetic minerals such as hematite and goethite have moderate positive magnetic susceptibility (MS) whereas water, quartz and feldspars have weak positive, respectively. Paramagnetic minerals like biotite and olivine have either low or no magnetic signals (Moritsuka et al., 2021). The magnetic mineralogy of soils and rocks depends on three important factors; the concentration of magnetic minerals, the shape and the size of magnetic spherules (Evans and Heller, 2003).

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A study in southeast France reported that magnetic characteristics of salt marsh soils polluted by fly ash settlement from the metal industry show enhanced magnetic particle concentration in the topsoil, and the results imply that pedogenesis, in addition to the pollution, is a contributing factor (Lecoanet et al., 2001).

Super-paramagnetic (SP) grains dominate the ferrimagnetic component of substantially enhanced surface soils with bigger stable single-domain/pseudo-single-domain (SSD/PSD) grains that may originate from magnetosomes and magnetic inclusions in a study performed in England (Dearing et al., 2001).

In Linfen City, China, researchers have examined tree leaves to learn more about their magnetic properties and how they affect the atmosphere (Yin et al., 2013). Results revealed that multi-domain (MD) magnetite-like minerals predominate the magnetic particles and that magnetic particle concentration and grain size decrease with increasing distance from an industrial area, demonstrating that the Linfen Steel Mill is the industrial area's main source of air particle pollution.

Many studies have been conducted to monitor the soil contamination in Iraq. A study performed by Abd Al-Qadir et al. (2023) to screen the hydrocarbon and heavy metal contamination of soils in Basra province using different techniques (investigating the study area using Google map, geological, hydrological, geographical investigation, laboratory tests including particle size analyses, pH, Electrical conductivity (EC), heavy metals, total petroleum hydrocarbon and digital image processing), they concluded that the studied area was heavily contaminated and needed to be monitored.

In Kirkuk province, a distinct study was conducted to investigate the geochemical effects of heavy metal contamination in agricultural lands. The findings revealed that the soil samples from different depths were only slightly or weakly contaminated with heavy metals, mainly cadmium (Ali et al., 2021; Awadh and Al-Hamdani, 2019). This study aims to investigate the magnetic mineralogy (magnetic carriers in soils) of agricultural lands from four different environments within the north of Al Muthanna Province, i.e., to examine the grain sizes of the magnetic minerals, correlate the magnetic susceptibility with heavy metals (HM) even whether there is a correlation between magnetic enhancement and heavy metals.

2. Materials and Methods

2.1. Study area

Four agricultural areas within Al Muthanna Province have been investigated; Rumaitha (RU), Samawa (SA), Warka’ (WA), and Swair (SR), with coordinates 31°30'51" N, 45°12'17" E, 31°18'52" N, 45°13'55" E, 31°28'23" N, 45°19'09" E, and 31°18'33" N, 45°22'14" E, respectively, (Fig. 1). The area is selected to examine their magnetic enhancements in soils and heavy metal accumulations and to investigate the correlation between them. Four groups of topsoil samples were collected as follows: Rumaitha, Samawa, Swair, and Warka’ (45, 45, 35, and 25), respectively. The samples were dried, stored in plastic bags, and transported for magnetic measurements at magnetic laboratory of Tübingen University, Germany.
B

Samawa Location
Legend
- Sample location
- World Imagery
Fig. 1. (A, B, C, and D) shows a location map showing the study area: A is for RU location; B is for SA location; C is for WK location; and D is for SR location.

2.2. Magnetic Measurements

The samples are prepared and magnetic measurements have been conducted in the magnetic laboratories, Department of Geosciences at Tubingen University, Germany. The samples were weighed and packed into 10 cm$^3$ plastic cylindrical containers before running the magnetic measurements.
Systematic measurements of soil magnetic susceptibility are carried out after collecting samples from four selected areas where top soils are likely to contain contaminants from different sources.

Magnetite is the most abundant ferrimagnetic mineral, and magnetic susceptibility is directly related to ferrimagnetic mineral concentrations. Magnetic susceptibility measurements of samples were done using an AGICO KLY-3 Kappa-bridge, such susceptibility was expressed in specific mass units \( (\chi, m^3 \cdot kg^{-1}) \). It is roughly inversely correlated with the concentration of paramagnetic minerals like iron-bearing silicates as well as ferrimagnetic minerals like iron oxides and hydroxides, iron sulphides, iron oxides and hydroxides. To calculate the grain-size distribution of the various sets of samples and concentration variations along profiles, additional magnetic parameters were performed; an AGICO MFK1-FA Kappa-bridge was used to measure frequency-dependent susceptibility \( (\kappa_{fd}, \%) \) with a peak field of 200 Ampere/meter (A/m) at two frequencies [976 Hz (F1) and 15616 Hz (F2)] to determine the possible presence of a super-paramagnetic (SP) mineral fraction (Hanesch and Petersen, 1999; Dearing et al., 1996).

Anhysteretic remanent magnetization (ARM) was induced using a 40 tesla (T) direct current (DC) bias field and a 100 millitesla (mT) peak alternating field and the intensity of the ARM was measured using a cryogenic 2G Enterprises superconducting magnetometer (2G-755R) (Jordanova and Jordanova, 1999; Jordanova et al., 1997). Isothermal remanent magnetization (IRM) was introduced using an MMPM9 pulse magnetizer. The IRM intensity was measured with a rotating Molspin magnetometer. To calculate the S-ratio using the Bloemendal equation, the saturation IRM (SIRM) at 1000 mT and an IRM-300 backfield in a 300 mT reverse field were both taken into account (Bloemendal et al., 1992). High temperature of magnetic susceptibility (k-T curves) for selected samples were done using the Kappa-bridge KLY3 equipped with a furnace and a CS-3 heating unit (maximum temperature of 700°C).

Finally, ferrimagnetic minerals in soils were identified, and potential sources of contamination were identified using bivariate analyses, which combine pairs of magnetic characteristics recorded at room temperature.

3. Results and Discussion

3.1 Magnetic Susceptibility

Magnetic susceptibility measurements were conducted to examine whether or not the sediments in the four agricultural areas are contaminated (i.e., to identify the potential source of contamination) due to the fact that the magnetic concentration of the signals will increase with increasing sediment contamination.

Box-whiskers of magnetic susceptibility are shown in Fig. (2). The figure shows that the highest quartile lies in RU, followed by SA, SR, and WK, respectively. The values of mass-specific magnetic susceptibility \( (\chi) \) range between 489.96 and 375.70 \( \times 10^{-8} \) \( m^3 \cdot kg^{-1} \) for RU, 480.40 and 260.80 \( \times 10^{-8} \) \( m^3 \cdot kg^{-1} \) for SA, 281.33 and 233.02 \( \times 10^{-8} \) \( m^3 \cdot kg^{-1} \) for SR and 234.89 and 147.51 \( \times 10^{-8} \) \( m^3 \cdot kg^{-1} \) for WK.

These different results may be the consequence of pedogenesis, which occurs when surface samples include varied amounts of organic matter. Although it appears that dust fallout is associated with topsoil susceptibility levels, pedogenesis may also be able to account for such large concentrations of magnetic materials (Maher, 1986). According to Thompson and Oldfield (1986), the soil around major cities and manufacturing areas may have higher susceptibility than other soil types.

3.2 Magnetic Parameters Ratios

There is a significant correlation between magnetic susceptibility, ARM and SIRM (Figs. 3 and 4). The comparatively high correlations show a small contribution to the magnetism of samples from magnetic minerals with paramagnetism and super-paramagnetism, and ferromagnetic minerals are a
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The same pattern exists with SIRM: SA shows the best correlation between $\chi$ and SIRM with a correlation coefficient of $R^2 = 0.92$ followed by WK with a correlation coefficient of $R^2 = 0.63$, while RU and SR show low correlation where correlation coefficients are $R^2 = 0.33$ for both of them.

Fig. 2. Box-Whisker of magnetic susceptibility ($\chi$) of samples collected from four study areas: RU, SA, SR, and WK.

Fig. 3. Correlation of magnetic susceptibility ($\chi$) versus susceptibility of Anhysteritic remanent magnetization ($\chi_{\text{ARM}}$) of samples collected from four study areas: (A) RU, (B) SA, (C) SR, and (D) WK.
We can discriminate between ferrimagnetic and anti-ferrimagnetic components using the ratio IRM-300 mT/ SIRM (S-ratio) (Bloemendal et al., 1992). According to Lecoanet et al. (2001), a ratio of more than 0.7 specifically denotes the predominance of ferrimagnetic components. The S-ratio lies between 0.91 and 0.92 for all the samples from the four studied areas and is above 0.7, which indicates the dominance of soft magnetic minerals like magnetite (Table 1).

### Table 1. Statistical analyses of the s-ratio for the four study areas

<table>
<thead>
<tr>
<th>Study area</th>
<th>Rumaitha (RU)</th>
<th>Samawa (SA)</th>
<th>Swair (SR)</th>
<th>Warka’ (WK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min.</td>
<td>0.91</td>
<td>0.91</td>
<td>0.92</td>
<td>0.92</td>
</tr>
<tr>
<td>Max.</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
<td>0.99</td>
</tr>
</tbody>
</table>

### 3.3 Frequency Dependent Susceptibility

Super-paramagnetic (SP) particles in the sediments can be determined depending on the fact that ultrafine-grained super-paramagnetic particles are sensitive to frequency-dependent magnetic susceptibility (κ_{FD} %) (Dearing et al., 1996). Table (2) shows the statistical analyses of κ_{FD} % for the four study areas.

### Table 2. Statistical analyses of frequency dependent susceptibility (κ_{FD} %) for the four study areas

<table>
<thead>
<tr>
<th>Study area</th>
<th>Rumaitha (RU)</th>
<th>Samawa (SA)</th>
<th>Swair (SR)</th>
<th>Warka’ (WK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min.</td>
<td>0.39</td>
<td>0.28</td>
<td>1.05</td>
<td>0.67</td>
</tr>
<tr>
<td>Max.</td>
<td>8.68</td>
<td>9.77</td>
<td>7.51</td>
<td>6.94</td>
</tr>
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</table>

The mass-specific magnetic susceptibilities values (χ) are given as follows: 142.35×10^{-8} m^3.kg^{-1} and 142.19×10^{-8} m^3.kg^{-1} corresponding to the minimum and maximum values of RU, 106.43×10^{-8} m^3.kg^{-1} and 112.37×10^{-8} m^3.kg^{-1} corresponding to the minimum and maximum values of
Concentrations of Cu, Cr, Ni, Cd, Hg, and Pb have been measured. The Pearson correlation between

It has been demonstrated that samples with high $\kappa_{FD} > 6\%$ carry a significant proportion of super-paramagnetic (SP) grains and occupy an envelope defined by limits that may define grains < 0.015 $\mu$m and > 0.015 $\mu$m, while values of $\kappa_{FD}$ percentage 5% are carrying stable-single domain (SSD) grains, however, samples with fine grains < 0.005 mm were dominated the SP fraction. As mentioned above, the particles show a mixture of SP and SD grains.

3.4 Temperature Dependent Susceptibility for Representative Samples

Magnetic minerals can be identified in the samples by measuring temperature-dependent susceptibility. The type of magnetic mineral can be determined by its Curie temperature using high-temperature-dependent magnetic characteristics (Ju et al., 2004). During the range of heating between 0 and 700 °C, the magnetic susceptibility of samples will drop dramatically if the temperature reached the Curie temperature of magnetic minerals. Representative samples (RU2, RU5, SA13, SA18, SR32, SR46, WK23 and WK27) have been chosen to display the high temperature-dependent susceptibility curves as shown in Fig. 5. All samples exhibit a reduction near the temperature of 580°C, reaching their baseline at about 580°C, which is the Curie temperature of magnetite in which by Thompson and Oldfield (1986) shows the existence of magnetite. When temperatures are lower than 500°C, cooling curves appear to be more susceptible than heating curves and such condition suggests that more magnetic minerals may be formed during heating (Hu et al., 2008).

![Fig. 5. Shows the thermo-magnetic analyses of representative samples from four areas: RU, SA, SR, and WK.](image)

4. Heavy Metal Analysis

Heavy metal (HM) contents have been determined for selected samples using XRF analysis. Concentrations of Cu, Cr, Ni, Cd, Hg, and Pb have been measured. The Pearson correlation between $\chi$
and HM contents is shown in Fig. 6. For RU samples, there is a weak positive correlation between the concentrations of Cu, Cd, Hg and Pb, except for Ni concentrations which show a negative correlation.

For SA, all samples show that the magnetic susceptibility is negatively correlated with the concentrations of Cu, Ni, Cd, Hg and Cr; the same is true for SR samples, which also show that the magnetic susceptibility is negatively correlated with the concentrations of Cu, Ni, Cd, Hg and Cr, while the best correlation is found in WK samples which show a good positive correlation between the concentrations of Cu, Ni, Cd, Hg, Pb and Cr. According to Pearson correlation analysis, magnetic susceptibility has a substantial negative correlation with heavy contents like Cu, Ni, Cd, Hg, Pb and Cr. This correlation results suggest that the areas could not be contaminated with heavy metals.

5. Conclusions

Magnetic mineralogy enhancements have been detected in four agricultural areas in Al Muthanna province; Rumaitha (RU), Samawa (SA), Swair (SR) and Warka’ (WK), due to the fact that sediments contamination enhances the magnetic susceptibility. The values of mass-specific magnetic susceptibility range between $489.96 \times 10^{-8}$ and $375.70 \times 10^{-8} \text{m}^3\cdot\text{kg}^{-1}$ for RU, $480.40 \times 10^{-8}$ and $260.80 \times 10^{-8} \text{m}^3\cdot\text{kg}^{-1}$ for SA, $281.33 \times 10^{-8}$ and $233.02 \times 10^{-8} \text{m}^3\cdot\text{kg}^{-1}$ for SR and $234.89 \times 10^{-8}$ and $147.51 \times 10^{-8} \text{m}^3\cdot\text{kg}^{-1}$ for WK, magnetite-like ferromagnetic minerals dominate the samples which show a mixture of super-paramagnetic and stable-single domain (SSD) grains. Heavy metals are not correlated with mass-specific magnetic susceptibility for Rumaitha, Samawa, Warka’ i.e., reveal negative correlation, except for Swair, which shows a good positive correlation.

The findings demonstrate a significant soil contamination in all four study sites, which will serve as a springboard for additional research on heavy metals in future projects.
Fig. 6. A, B, C, D Show the correlation between magnetic susceptibility (χ) and concentration of heavy metal (HM) in representative samples from four areas: (A) RU, (B) SA, (C) SR, and (D) WK.

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References


