

## Compositional variation of soils from Cabo Mondego (Portugal): new data to support environmental studies

## Variação composicional dos solos do Cabo Mondego (Portugal): novos dados de suporte para estudos ambientais

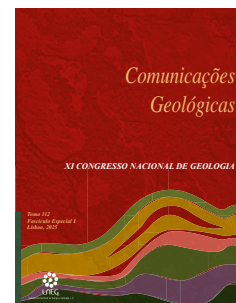
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**Abstract:** This study aims to determine physicochemical parameters of soils from Cabo Mondego area (Central Portugal), allowing the establishment of the natural background to serve as a reference to identify variations resulting from industrial activities. Forty-three surface soil samples were collected near the deactivated industrial complex and another seven in more distant areas. The soils present a predominantly sandy texture and pH values in the alkaline range, which reflects the geological signature. The soils electrical conductivity of five samples present values above the Portuguese reference value for contaminated soils. The anomalous values measured in a sample (CM-S3) were related to the presence of coal mining wastes located nearby. From the determined elements, the most abundant is Ca reflecting the limestone rocks composition, and a lithological control was identified in the spatial analysis, where higher concentrations of Fe and Al are coincident to the spatial occurrence of carbonaceous complex and limestones.

**Keywords:** Cabo Mondego, background, geochemical composition, spatial analysis, contamination.

**Resumo:** Este estudo tem como objetivo determinar parâmetros físico-químicos dos solos da área do Cabo Mondego (Centro de Portugal), permitindo o estabelecimento do fundo natural para servir como referência para identificação de variações composicionais resultantes de atividades industriais. Quarenta e três amostras de solos superficiais foram coletadas próximas ao complexo industrial desativado e outras sete em áreas mais distantes. Os solos apresentam predominantemente textura arenosa e valores de pH na faixa alcalina, o que reflete a assinatura geológica local. Os dados de condutividade elétrica do solo mostram que cinco amostras apresentam valores superiores aos de referência para solos contaminados em Portugal. Os valores anómalos medidos na amostra CM-S3 estão relacionados com resíduos de mineração de carvão localizados nas proximidades. Dos elementos analisados, o mais abundante é o Ca, refletindo a composição de rochas calcárias, e foi identificado um controle litológico na análise espacial, em que amostras com maiores concentrações de Fe and Al estão localizadas na área de ocorrência do complexo carbonoso e calcários.

**Palavras-chave:** Cabo Mondego, fundo natural, composição geoquímica, análise espacial, contaminação.

### 1. Introduction

The soils from Cabo Mondego are exceptionally relevant from an environmental standpoint as it is a highly significant territory of natural and cultural interest in Portugal. The area was classified as a Portuguese Natural Monument in 2007 (Decreto Regulamentar No. 82/2007) according to important research findings on its stratigraphic and paleontological record, which allowed the establishment of a Global Boundary Stratotype Section and Point (GSSP) for the base of the Bajocian stage (Middle Jurassic) (Henriques *et al.*, 1994) and an Auxiliary Boundary Stratotype Section and Point (ASSP) for the base of the Bathonian stage (Middle Jurassic) (Fernández-López *et al.*, 2009 a, b). The investigations have also revealed other significant natural aspects within the geodiversity of the site (Rocha *et al.*, 2014), which are particularly relevant on the ongoing Atlantic Geopark Project (Vaz *et al.*, 2023), aiming the integration of the Cabo Mondego territory into the United Nations Educational, Scientific and Cultural Organization (UNESCO) Global Geoparks Network.

From another perspective, Cabo Mondego also reveals a long history of extractive and industrial activities, recognized for being one of the first coal mines in Portugal (Pinto *et al.*, 2014) and for hosting a significant industrial complex for over a century, comprising several types of factories, with emphasis on the production of hydraulic lime and cement (Goulão, 2016). All activities of this industrial complex are deactivated since 2013. The cement and lime industries are known to generate a great diversity of contaminants including potentially toxic elements (PTE) and organic compounds as polycyclic aromatic hydrocarbons, which impact the environment near these places (Wang *et al.*, 2018; Parlak *et al.*, 2023). The fossil fuel combustion necessary to supply high temperature furnaces, in parallel with the processing of raw materials (quarrying, grinding, etc.) are considered the main sources of the contaminants release (Cutillas-Barreiro *et al.*, 2016). Therefore, the study of natural characteristics of the soils in the Cabo Mondego area becomes essential to evaluate the environmental impact caused by past activities in the industrial complex along adjacent locations.

The main goal of this study is to determine physicochemical parameters in soils from the Cabo Mondego area, establishing the local natural background characteristics to provide a reference for detection of variations caused by industrial activities. This knowledge is crucial to distinguish sources of potential soil contamination in future studies, when analyzed together with the concentration of PTE or other critical environmental parameters.

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## 2. Geological setting

The lithologies that make up the geological setting of the study area are composed of Jurassic deposits from the northern sector of the Lusitanian Basin, which hold significant scientific value due to paleontological and stratigraphic records (Rocha *et al.*, 2014). The sedimentary record of the Middle Jurassic is exposed in the northern sector of the study area and corresponds to the Cabo Mondego Formation, composed of limestone, marly limestone, and marls (Figura 1). The Upper Jurassic succession, which outcrops in the rest of the study area, is made up of limestones and coal layers corresponding to the carbonaceous complex of the Vale Verde Formation (targeted for coal exploitation in the Cabo Mondego mine), and sandstones from the Boa Viagem Formation (Rocha *et al.*, 1981; Pena dos Reis, 2006; Trincão *et al.*, 2018).

Soils of the area are classified as calcic cambisols, according to

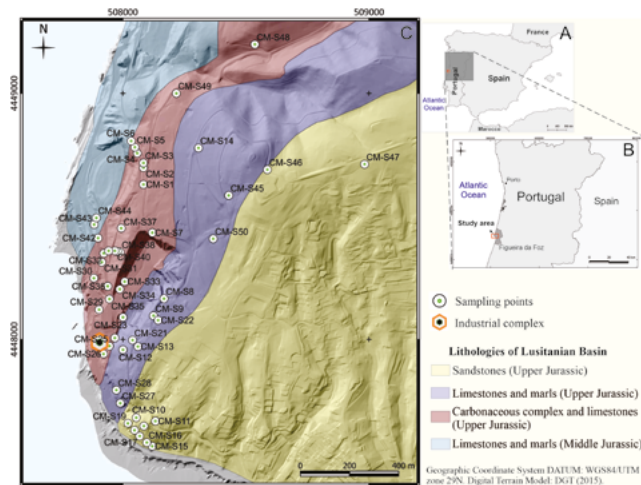


Figure 1. Location map of the study area within Iberian Peninsula and Portugal (A and B, respectively) and geological map with identification of sampling points (C) (modified from Rocha *et al.*, 1976). Digital Terrain Model (DGT, 2015).

Figura 1. Mapa de localização da área de estudo na Península Ibérica e em Portugal (A e B, respetivamente). Mapa geológico com identificação dos pontos de amostragem (C) (modificado de Rocha *et al.*, 1976). Modelo Digital de Terreno (DGT, 2015).

the Soil Map of Portugal (Cardoso, 1974). They are characterized by slightly developed horizon differentiation in the subsoil and strongly drained with incipient formation time soils, in this case categorized as a mineral-origin soils with an ochric A horizon and a calcic horizon (FAO, 1974; FAO, 2015).

## 3. Material and methods

A total of 50 surface soil samples were collected for this study. From these, 43 samples were taken in the vicinity of the deactivated industrial complex, and 7 in more distant areas (CM-S14, CM-S45, CM-S46, CM-S47, CM-S48, CM-S49 and CM-S50) (Figura 1). Samples preparation in the laboratory included drying, sieving to obtain the fraction below 2 mm, and crushing.

Soil grain size analysis was performed using 2 mm and 0.063 mm mesh sieves to separate the fractions of mud, sand and gravel. The pH and electrical conductivity (EC) were measured according to ISO 10390 (2021) and ISO 11265 (1994), respectively. These analyses were performed at the laboratories of the University of Coimbra (Portugal). Total organic carbon (TOC) was determined using the Eltra CS-530 equipment with a total inorganic carbon (TIC) module analyzer at the

University of Silesia (Poland), calibrated utilizing the Eltra standards. The TOC was calculated as the difference between the total carbon and TIC. The chemical composition of major elements was analyzed by inductively coupled plasma emission spectrometry/mass spectrometry (ICP-ES/MS) at Bureau Veritas Mineral Laboratories (Canada).

## 4. Results and discussion

Table 1 presents the minimum, maximum, mean, and standard deviation values of the parameters determined in the soil samples. Based on the obtained results, patterns in the soils chemical composition were sought through statistical analysis.

Based on the grain size analysis results, the soils showed a predom-

Table 1. Minimum, maximum, mean, and standard deviation (SD) values of granulometric fractions (%), TOC (%), pH, EC ( $\mu\text{S}/\text{cm}$ ) and concentration of major elements (%) determined in the soil samples.

Table 1. Valores mínimos, máximos, média e desvio padrão das frações granulométricas (%), carbono orgânico total (COT) (%), pH, condutividade elétrica (CE) ( $\mu\text{S}/\text{cm}$ ) e concentração em elementos maiores (%) determinados nas amostras de solo.

Parameters	Minimum	Maximum	Mean	SD
Gravel	1.49	57.9	25.8	12.1
Sand	40.8	79.2	66.4	9.40
Mud	0.55	25.5	7.79	5.36
TOC	0.54	37.0	5.11	5.44
pH	4.45	8.61	7.90	0.84
EC	52.4	1627	369	360
Ca	0.13	26.9	7.68	6.71
Al	2.56	8.55	4.30	1.27
Fe	0.47	4.95	1.90	0.81
K	0.48	2.55	1.51	0.51
Mg	0.08	0.57	0.35	0.12
Na	0.07	0.55	0.22	0.12
Ti	0.10	0.36	0.19	0.06
S	0.02	1.04	0.15	0.17
P	0.01	0.06	0.03	0.01

inance of sand (Tab. 1). Forty-five samples have sand content above 50%. In the ternary diagram for textural classification of sediments based on gravel, sand, and mud content proposed by Folk *et al.* (1970), most of the samples are plotted in the gravelly sand and gravelly muddy sand categories (Figura 2). There are also few samples in the categories of slightly gravelly muddy sand, slightly gravelly sand, and muddy sand. On the other hand, 5 samples showed a predominance of gravel; these samples are from geographically dispersed sampling points, namely, in the vicinity of the industrial complex (CM-S30, CM-S31, and CM-S38) and to the north of the industrial complex (CM-S5, CM-S48). These samples are classified in the sandy gravel category on the textural diagram since they have sand content above 40%.

The grain size data was compared with data provided by the *Infosolo* database from National Institute of Agrarian and Veterinary Research of Portugal (Ramos *et al.*, 2017), where sand content between 50% and 70% is defined for the Cabo Mondego area. The measured values for this study range between 40.83% and 79.18%, which is in accordance with the referred database (Ramos *et al.*, 2017).

The TOC values range from 0.54% to 37.0%, with a mean value of

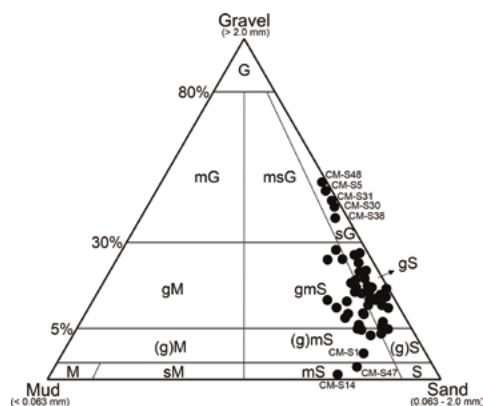


Figure 2. Textural classification of gravel-bearing sediments proposed by Folk *et al.* (1970). G – Gravel; mG – muddy gravel; msG – muddy sandy gravel; sG – sandy gravel; gM – gravelly mud; (g)M – slightly gravelly mud; M - mud; sM – sandy mud; gmS – gravelly muddy sand; gS – gravelly sand; (g)mS – slightly gravelly muddy sand; (g)S – slightly gravelly sand; mS – muddy sand; S – sand.

Figura 2. Classificação textural de sedimentos contendo cascalho proposta por Folk *et al.* (1970). G – Cascalho; mG – cascalho lamacento; msG – cascalho arenoso/lamacento; sG – cascalho arenoso; gM – lodo cascalhoso; (g)M – lodo ligeiramente cascalhoso; M - lodo; sM – lodo arenoso; gmS – areia lamacenta/cascalhosa; gS – areia cascalhosa; (g)mS – areia lamacenta ligeiramente cascalhosa; (g)S – areia ligeiramente cascalhosa; mS – areia lamacenta; S – areia.

5.11% (Tab. 1). The samples with values below 5% represent the majority, totaling 33 out of the 50 samples. Only 4 samples presented TOC values above 10%, 3 of them within the range between 10.8% and 12.8%. Sample CM-S3 shows an anomalous value of 37.0%, which is explained by the fact that sample was collected near coal mining wastes identified in the field. Figure 3A shows the spatial distribution of TOC values in the sampled area.

The soils presented a pH ranging from 4.45 to 8.61 (Tab. 1). According to the pH classification by the Food and Agriculture Organization (FAO) (Jahn *et al.*, 2006), values between very slightly alkaline and moderately alkaline ranges (7.10 to 8.61) were recorded in 44 samples across the entire sampled area. Values from the strongly acid to neutral ranges (4.45 to 7.00) were measured in 6 samples, including 5 samples from more distant areas and the sample CM-S3, collected close to the coal mining wastes (Figura 3B). Generally, the soils of Cabo Mondego are classified as strongly acidic to moderately alkaline, with a predominant slightly

alkaline nature, primarily reflecting the geologic signature of the alkaline limestone lithology that predominate in the local geology.

The pH values were also compared to the data provided by *Infosolo* database (Ramos *et al.*, 2017), where soils in the region are characterized by a pH ranging from 4.6 to 5.5, within the acidic range. This data is consistent with the pH of only one sample measured for this study (CM-S47). Most of the other samples (88% of the total) were measured within the alkaline range. When analyzing the sampling locations used to create the pH map by *Infosolo* database (Ramos *et al.*, 2017), it is noticeable that there are few sampling points close to the study area (two points in the village of Quiaios with an average distance of 6 km from the study area and three points in the city of Figueira da Foz with an average distance of 11 km from the study area), which may explain the differences with this study.

The EC values range from 52.4  $\mu\text{S}/\text{cm}$  to 1627  $\mu\text{S}/\text{cm}$  (Tab. 1). The lower values, between 52.4  $\mu\text{S}/\text{cm}$  and 570  $\mu\text{S}/\text{cm}$ , were measured in 45 samples (90% of the total). The samples with values above 570  $\mu\text{S}/\text{cm}$ , which is the reference value to identify potential contamination problems in environmentally sensitive soils set by the Portuguese Environment Agency (APA, 2019), amount to a total of 5. These samples are in the southern sector of the study area (CM-S17), in the central sector, very close to the deactivated cement and lime industrial complex (CM-S24, CM-S25 and CM-S33), and in the northern sector of the study area (CM-S3) (Figura 3C).

The EC data are an indicator of the amount of soluble chemical species in the soils and show relatively little variation in a large portion of the samples. Values above the APA reference for environmentally sensitive soils should be carefully considered as an indicator of possible contamination.

In terms of the major elements, the chemical composition of the samples included mean concentration above 1% for Ca, Al, Fe, and K, followed by mean concentration below 1% for Mg, Na, Ti, S, and P (Tab. 1). From the determined major elements, Ca is the most abundant element, reaching values in the range of 20% in some samples (Figura 4), which reflects the geological characteristics of the study region where predominates limestone lithologies. The highest S value (1.04%) was recorded in the sample CM-S3, collected close to the coal mining wastes, being nearly three times higher than the second-highest S value of sample CM-S4 (0.36%), located adjacent to CM-S3. The higher concentration of S in these samples is most probably related to the coal mining waste material located nearby. Studies about the coal from Cabo Mondego revealed a total S of 6.14% (Lemos de Sousa

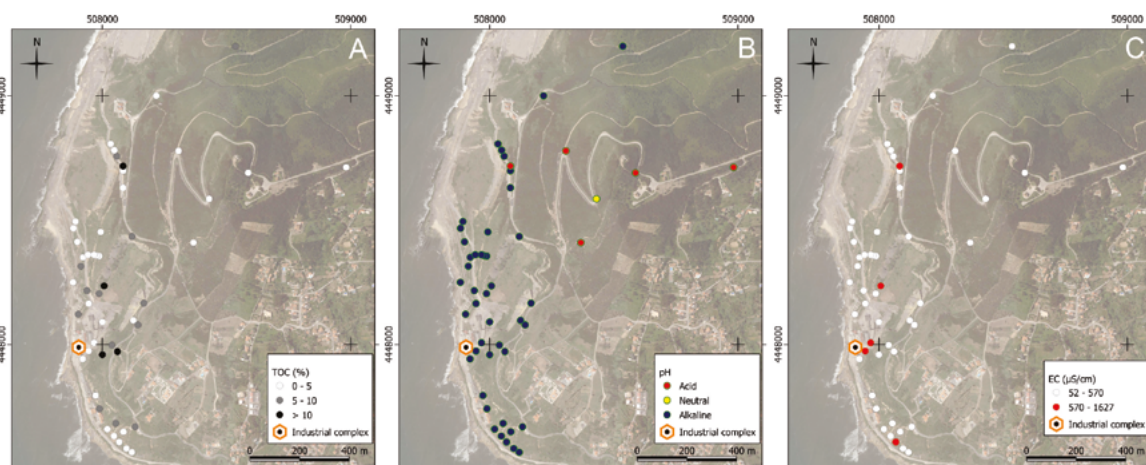


Figure 3. Spatial distribution of TOC (A), pH (B) and EC (C) in the studied soil samples.

Figura 3. Distribuição espacial de COT (A), pH (B) e CE (C) nas amostras de solo estudadas.

*et al.*, 2010). Consequently, the chemical composition of the waste material reflects the coal composition. After deposition of the waste material, weathering processes promote the release of S to adjacent soils.

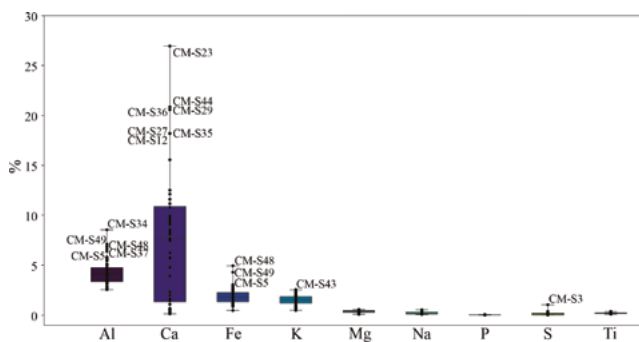


Figure 4. Boxplot diagram showing the ranges of concentrations of major elements in the soil samples.

Figura 4. Diagrama Boxplot mostrando as faixas de concentrações dos elementos maiores nas amostras de solo.

Since the data do not present a normal distribution, Spearman correlation analysis was calculated. Data from sample CM-S3 were excluded to reduce the results bias, since this sample has anomalous characteristics already addressed. The coefficients revealed strong or very strong positive correlation between Al-Fe (0.91), Al-Ti (0.82), and Fe-Ti (0.71) (Tab. 2). These elements may accumulate specially in the clay fraction of the soil due to their low mobility and are associated with oxides resulting from natural weathering process (Sposito *et al.*, 2008; Santos *et al.*, 2020). Higher values of Al and Fe are noticed in the samples of the area mapped as Carbonaceous Complex and Limestones (Figura 5) and may represent a lithological control of these elements' concentration on these specific soils. It is worth noting that samples with higher concentrations of Ti and S are also in the same lithology, despite most concentrations of these elements are in quantities below 1%. The lowest mean concentration of Ca appears among the samples located in the sandstone lithology. The other major elements occur in varying concentrations, without an identified spatial pattern for the study area.

Ca-Mg (0.81) correlation coefficient is also very strong (Tab. 2) due to  $\text{CaCO}_3$  and  $\text{CaMg}(\text{CO}_3)_2$  accumulation is the most important soil forming process in this area, controlled by a limestones and marls lithologies signature. On the other hand, there are significant negative correlations between Ca-K (-0.73) and Ca-Na (-0.57). The dominant presence of Ca in calcareous soils may affect the availability of K and Na, since  $\text{Ca}^{+2}$  is more easily adsorbed than  $\text{Na}^+$  and  $\text{K}^+$  on the solutions (Taalab *et al.*, 2019).

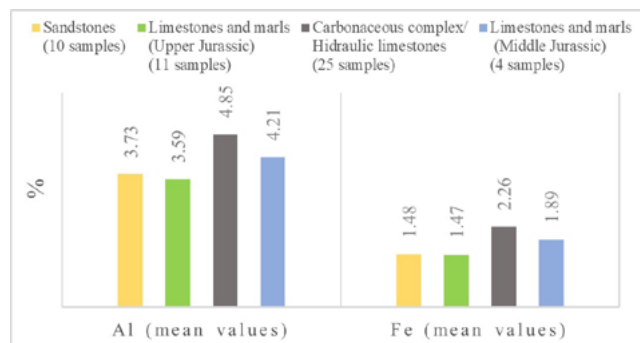


Figure 5. Fe and Al mean concentrations in each lithology mapped for the study area.

Figura 5. Concentrações médias de Fe e Al em cada litologia mapeada para a área de estudo.

5. Conclusions

In general, the soils in the study area have a sandy texture, TOC values below 5%, alkaline pH and show slight variations in EC values. Most of EC values are below the reference values set by APA for environmentally sensitive soils. The anomalous values of TOC, pH, EC and S measured in sample CM-S3 are related to the presence of coal mining wastes identified in the field.

The geochemical composition of soils regarding major elements and the correlation coefficients reveals the lithological control dominated by limestones/marls and it is in accordance with the occurrence of cambisols described for the region.

The results regarding the physicochemical characterization

Table 2. Spearman's linear correlation matrix for physicochemical parameters (TOC, pH, and EC) and major elements. Bold values are significant at  $p < 0.05$ .

Tabela 2. Matriz de correlação de Spearman para parâmetros físico-químicos (COT, pH e CE) e elementos maiores. Valores em negrito são significativos em  $p < 0.05$ .

	TOC	pH	EC	Ca	Al	Fe	K	Mg	Na	Ti	S	P
TOC	1											
pH	<b>-0.45</b>	1										
EC	-0.06	0.13	1									
Ca	0.14	<b>0.28</b>	<b>0.51</b>	1								
Al	-0.25	<b>0.34</b>	<b>0.31</b>	-0.01	1							
Fe	-0.14	<b>0.39</b>	<b>0.32</b>	0.18	<b>0.91</b>	1						
K	<b>-0.45</b>	0.00	-0.23	<b>-0.73</b>	0.19	-0.01	1					
Mg	0.06	<b>0.36</b>	<b>0.67</b>	<b>0.81</b>	0.19	<b>0.32</b>	<b>-0.44</b>	1				
Na	-0.14	-0.16	-0.26	<b>-0.57</b>	0.14	0.00	<b>0.53</b>	<b>-0.59</b>	1			
Ti	<b>-0.42</b>	<b>0.32</b>	0.02	<b>-0.30</b>	<b>0.82</b>	<b>0.71</b>	<b>0.43</b>	-0.07	<b>0.38</b>	1		
S	<b>0.49</b>	-0.03	<b>0.50</b>	<b>0.75</b>	-0.05	0.15	<b>-0.69</b>	<b>0.62</b>	<b>-0.36</b>	<b>-0.35</b>	1	
P	<b>0.38</b>	0.10	<b>0.51</b>	<b>0.70</b>	0.05	0.19	<b>-0.52</b>	<b>0.69</b>	<b>-0.46</b>	-0.23	<b>0.66</b>	1

may contribute to the *Infosolo* database in the future, since few data regarding soils were found in the region surrounding the study area. More than that, these findings will be highly valuable as a contribution to a deeper understanding of vulnerabilities associated with the Natural Monument, to identify environmental issues arising from past industrial activities, as well to assist the area requalification actions to be implemented where the industrial complex is located. The knowledge acquired will enable informed decision-making based on scientific evidence, which is particularly relevant within the context of the ongoing Atlantic Geopark project, which aims to integrate the territory into the UNESCO Global Geoparks Network.

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