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THERAPEUTIC MUD OCCURRENCES IN GREECE: MINERALOGICAL AND GEOCHEMICAL COMPOSITION OF THE SAGIADA MUD (THESPROTIA PREFECTURE)

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Abstract

The current study presents the preliminary results of the mineralogical and geochemical characterization of the Sagiada mud (Prefecture of Thesprotia), which is considered as one of the most representative therapeutic mud occurrences in Greece. This work is part of a bigger project, conducted by IGME (Athens, Greece), for the characterization of the Greek therapeutic mud deposits. The mineralogical composition was determined using X-Ray Diffraction (XRD), Differential Thermal Analysis (DTA), optical microscopy and Scanning Electron Microscopy (SEM). The main mineral phases of the Sagiada mud are quartz, feldspars, clay minerals such as illite, kaolinite, chlorite and vermiculite, and calcite accompanied by minor phases such as halite and pyrite. Traces of muscovite and gypsum were also identified. Geochemical analyses were performed using X-Ray Fluorescence (XRF) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) for the determination of major and trace element content, respectively. Compared to Spanish peloids and European floodplain sediments, the Sagiada mud reveals an analogue chemical composition.

Keywords: *mud-therapy, clay minerals, mineralogy, geochemistry.*

Περίληψη

Η παρούσα εργασία αποτελεί μία πρώτη προσπάθεια χαρακτηρισμού των ορυκτολογικών και γεωχημικών χαρακτηριστικών του πηλού της Σαγιάδας (Νομός Θεσπρωτίας), όπου εντοπίζεται μία από τις πιο αντιπροσωπευτικές εμφανίσεις ιαματικού πηλού στην Ελλάδα. Η μελέτη αυτή αποτελεί τμήμα ενός ευρύτερου ερευνητικού προγράμματος που υλοποιείται από το Ι.Γ.Μ.Ε. για το χαρακτηρισμό των ελληνικών ιαματικών πηλών. Όπως προέκυψε από τα αποτελέσματα της περιθλασιμετρίας ακτίνων–Χ (XRD), διαφορικής θερμικής ανάλυσης (DTA), οπτικής μικροσκοπίας και ηλεκτρονικής μικροσκοπίας σάρωσης (SEM), ο πηλός της Σαγιάδας συνίσταται από χαλαζία, αστρίους, αργιλικά ορυκτά, όπως ιλλίτης, καολινίτης, χλωρίτης και βερμικουλίτης, και ασβεστίτη, καθώς και από δευτερεύοντα ορυκτά, όπως αλίτης και σιδηροπυρίτης. Επίσης εντοπίστηκαν ίχνη από μοσχοβίτη και γύψο. Οι χημικές αναλύσεις πραγματοποιήθηκαν με τη χρήση φασματομετρίας ακτίνων–Χ (XRF) και φασματομετρίας μάζας επαγωγικά συζευγμένου πλάσματος (ICP-MS), για τον προσδιορισμό των συγκεντρώσεων των πηλών σε κύρια στοιχεία και ιχνοστοιχεία, αντίστοιχα. Σε σύγκριση με πηλοειδή από την Ισπανία και το εύρος τιμών των

πλημμυρικών ιζημάτων από την Ευρώπη, ο φυσικός πηλός της Σαγιάδας παρουσιάζει ανάλογη σύσταση.
Λέξεις κλειδιά: *πηλοθεραπεία, αργιλικά ορυκτά, ορυκτολογία, γεωχημεία.*

1. Introduction

The empirical use of clays and muds for therapeutic purposes is known since antiquity, with the most common uses to be the ingestion and skin application in the form of patches, cataplasms or mud-baths (Gomes and Silva, 2007). In ancient Greece, mud materials, known as medical earths (Terrae), were often named after their origins. They were used as antiseptic cataplasms to cure skin afflictions or as a cure for snake-bites (Carretero, 2002). Example of medical earths is the Terra Lemnia, from Lemnos Island in North Aegean Sea, which was known as a pharmaceutical product from antiquity until the 19th century.

Nowadays, clay minerals, such as smectites, palygorskite, kaolinite and talc, are used in pharmaceutical formulations as well as in spa-centers and aesthetic medicine for therapeutic purposes (Carretero, 2002). According to Veniale *et al*. (2007), pelotherapy, which is the use of thermal muds for muscle-bone-skin pathologies treatment, is also applied for wellness and relaxation. Though, since the natural reserves of thermal muds are limited, many spa-centers produce the thermal mud by "maturation" (Veniale *et al*., 2007). Maturation is a process of mixing natural sediments potentially interesting for therapeutic or cosmetic purposes, with mineral water or seawater, or particularly with medicinal mineral water (Gomes *et al*., 2013). According to Gomes *et al*. (2013), mud is also applied in the natural environment, where it occurs, in the form of mud-packs, mud-baths or facial masks, sometimes in an empirical way, for healing or cosmetic purposes, in a practice so-called "mud-therapy". In Greece, mud-therapy is widely known and applied, especially during the summer period.

The current study is part of a project for the characterization of Greek mud deposits, used for therapeutic purposes. The project is funded by the European Union (NSRF 2007-2013, code 350913). In the framework of this project, mineralogical, geochemical and physicochemical analyses were carried out at the Institute of Geology and Mineral Exploration (IGME), Athens, Greece, in order to assess the suitability of Greek muds for therapeutic use. The studied areas include the Sagiada mud occurrences (Thesprotia, Epirus), the mud sediments of the Aetolikon - Messolonghi lagoon, local occurrences at Western Greece (such as Kyllini, Kaiafas Lake, Mytikas Xiromero and places of interest in Ambracian Gulf), as well as Argos (Eastern Peloponnese), Aedipsos (Evia Island) and the Aegean Islands Milos, Kos and Lemnos. An integrated database was designed and developed, in order to present general information, as well as the physicochemical, mineralogical and chemical properties of the studied mud occurrences. This study enhances the importance of these natural resources for local communities, on the base of potential exploitation through the development of medical tourism, entrepreneurship, and the use of natural reserves as raw material for spa-centers. In addition to the above, some other locations for mud-therapy in Greece are Krinides (Kavala Prefecture), Pikrolimni Lake (Kilkis Prefecture), Kavasila (Ioannina Prefecture), Vromolimni Lake of Methana (Attica Prefecture), Santorini Island, Amynteo (Florina Prefecture), Samos Island, Astros (Arkadia Prefecture) and the Anargiroi basin (Florina Prefecture) (Aggelidis, 1990).

Herein the case study of the Sagiada mud is presented, as it is considered one of the most representative natural therapeutic muds in Greece and is traditionally used for mud-therapy, both in-situ and in a local Mud-Therapy Center, after a beneficiation process. According to a previous study of the Sagiada mud, it is designated safe for use due to the restricted mobility of trace elements in water solution (Mitrakas, 2009). The present work examines in detail the mineralogical composition of the natural mud, the clay mineral content and the chemical composition.

2. Geological Setting

The study area is located in the Sagiada Bay of Thesprotia, NW Greece (Figure 1), a region that geotectonically belongs to the Ionian Zone.

Specifically, the area consists of sediments of Neogene to Quaternary age, including marls and recent alluvial deposits, mainly originate from the Thyamis (Kalamas) River. The lagoon environment that is formed extends in a SSW direction from the Sagiada settlement to the most recent deltaic deposits of Thyamis River, which is approximately 5 km far.

The Alpine basement outcropping in the area consists of a sedimentary sequence, which overlies the formation of Permo-Triassic evaporites. The base of this sequence consists of neritic limestones and dolomites, while the upper parts of Jurassic to Eocene age, consist of semi-pelagic to pelagic formations such as limestones, schists with *Posidonia* and cherts. Flysch sediments were deposited from Late Eocene to Early Miocene (Aquitanian); consist of sandstones, siltstones and marls (Perrier and Koukouzas, 1969; Mountrakis, 2010).

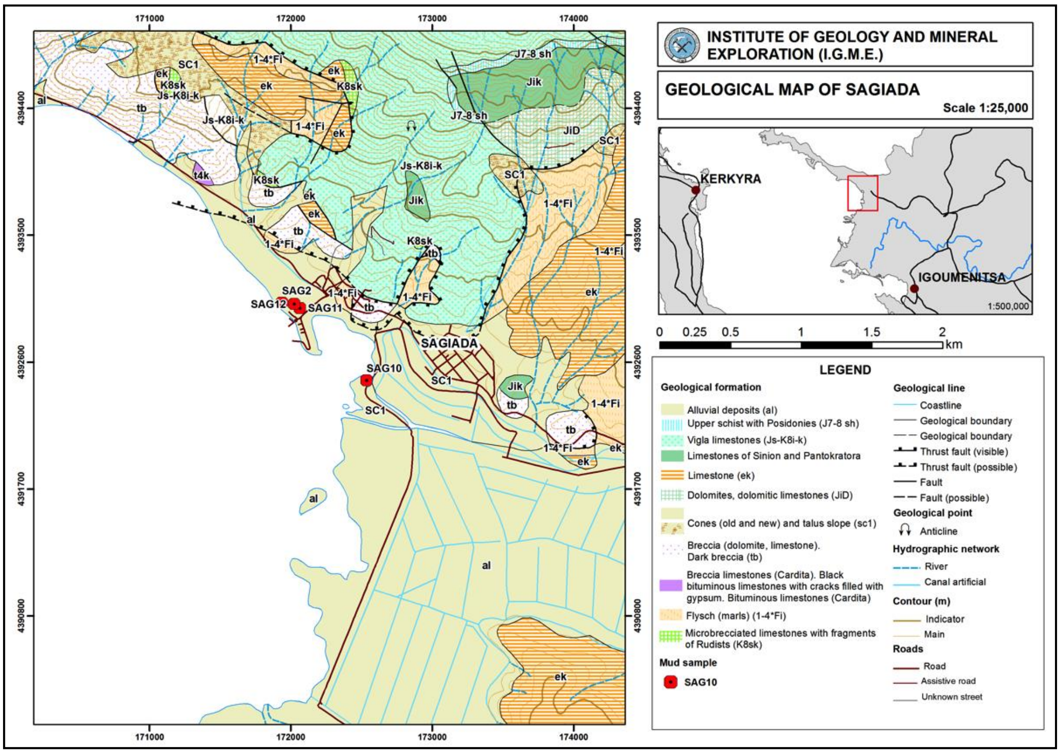

Figure 1 - Geological map of Sagiada (modified after Perrier and Koukouzas, 1969).

3. Materials and Methods

Six core sediment samples, approximately 2 kg each and up to 70 cm depth, were collected for mineralogical and geochemical analysis. The sampling locations are presented in Figure 1. The samples were dried, homogenized and pulverized using an agate mill at -200 mesh. The mineralogical composition was determined by four analytical techniques, such as X-Ray Diffraction (XRD), Differential Thermal Analysis (DTA), transmitted-light optical microscopy and Scanning Electron Microscopy (SEM), carried out at the Laboratories of Mineralogy and Petrology of the Institute of Geology and Mineral Exploration (IGME). X-Ray Diffraction analyses were performed using a Panalytical X' Pert PRO diffractometer with CuKα radiation and the scanning area covered the interval 2θ 3-70°, with a scanning step of 0.03° and a step time of 3 s. Clay minerals were identified on the bulk samples (2θ=3-60°), as well as on the samples after treatment with ethylenoglycole (2θ=3-30°) and after heating at 490 °C for 2 h (2θ=3-30°), with a scanning step of 0.03° and a time step of 3 s (Gee and Bauder, 1986; Moore and Reynolds, 1997). The mineral phases were identified with the use of EVA® software and semi-quantitative determination was performed using the X-Pert High Score software provided by Bruker. The DTA analyses were carried out with a DTG-60AH, in temperature range 30-1100 °C and step time 10 °C /min, using N gas with 50 ml/min flow. The optical microscopy was performed using a Zeiss AXIOSKOP 40 polarized-light microscope and the Scanning Electron Microscopy using a JEOL-JSM 5600, with associated Energy Dispersive Spectroscopy analyzer (EDS). Major element composition was determined by X-Ray Fluorescence (XRF) at the Laboratories of Mineralogy and Petrology of IGME, using a S4 Pioneer Bruker AXS (for SiO_2, TiO_2, Al_2O_3, MnO, MgO, CaO, Na_2O, K_2O, P_2O_5, Fe_2O_3 and SO_3). Trace element concentrations were determined in solutions, which were prepared after open vessel digestion with Aqua Regia, applying Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) at the Analytical Laboratories of IGME (for As, Ba, Be, Cd, Co, Cr, Cs, Cu, Mo, Ni, Pb, Sb, Se, U, V and Zn). The Loss on Ignition was measured after heating the samples at 1000 °C for 3 h, at the Analytical Laboratories of IGME.

4. Discussion and Results

The studied material, a natural mud from the Sagiada lagoon, represents sediments used for therapeutic purposes both in the natural environment and in a local Mud-Therapy Centre, close to the Sagiada settlement. The studied samples originate mainly from the local Mud-Therapy Centre, as well as from distal places to the SSW (Figure 1).

4.1. Mineralogical Composition

The semi-quantitative determination of the mineral matter of the Sagiada natural mud reveals that quartz (28-33 wt.%), feldspars (6-14 wt.%), clay minerals such as illite (10-17 wt.%), kaolinite, (3-7 wt.%), chlorite (4-6 wt.%) and vermiculite (4-11 wt.%), and calcite (17-26 wt.%) are the main mineral phases (Table 1). Halite (2-3 wt.%) and pyrite (1-3 wt.%) constitute minor phases (Table 1). Clay mineral identification was carried out especially using X-Ray Diffraction (XRD) in the clay fraction of the samples (Figure 2), as well as using Differential Thermal Analysis in the bulk samples (DTA) (Figure 3). Carbonate components occur in the form of cryptocrystalline aggregates (Figure 4a); whereas pyrite is often found in the form of framboids (Figure 4b). Traces of muscovite and gypsum were observed by transmitted-light microscopy.

In the study area, quartz, clay minerals (illite, chlorite) and feldspars have a clastic origin (Drees *et al*., 1989; Huang, 1989), deriving from the weathering and erosion of the surrounding Neogene and Quaternary rocks. Vermiculite and kaolinite are formed from the weathering of mica and chlorite and rarely by feldspars alteration (Paquet and Clauer, 1997). Calcite has clastic or authigenic origin (Doner and Lynn, 1989; Paquet and Clauer, 1997) from Ca-rich waters originated from limestone and evaporite dissolution. Halite may have clastic and/or authigenic origin or derive from the sea spray (Doner and Lynn, 1989; Paquet and Clauer, 1997). Pyrite in coastal mires is usually found in the form of framboids. Their formation is due to the reduction of the seawater sulphates and the reaction of the H_2S with Fe-ions, which derive from detrital material entering the mire during flood episodes (Dellwig *et al*., 2001).

4.2. Chemical Composition

The major and trace element composition is presented in Table 2 along with the compositional range of Spanish peloids (Carretero *et al*., 2010; Carretero *et al*., 2014), and floodplain sediments according to the Geochemical Atlas of Europe (FOREGS, 2005). The major element concentrations display a significant resemblance, as shown in Figure 5, except from CaO, which ranges between 10.46 and 17.39 wt.% and SO_3, which ranges between 0.93 and 2.16 wt.%. Compared to the

Spanish peloids, the Sagiada mud has a typical composition. Regarding trace element content, Ba, Ni, Cr, Zn and V were the most abundant elements (above 100 mg/kg), whereas Cu, Co, Pb, As, Mo, U, Be, Cd and Sb occur in lesser amounts. Compared to the Spanish peloids, the Sagiada mud is enriched in Ni and Cr and slightly in V and Co, but their concentrations seem reasonable compared to the compositional range of the floodplain sediments from Europe (FOREGS, 2005). Compared to floodplain sediments from Europe (FOREGS, 2005), the Sagiada mud occurs slightly enriched in SO_3. Although, background element concentration is essential in order to assess their origin.

Table 1 - Mineralogical composition of the Sagiada natural mud, as identified using X-Ray Diffraction analysis (in wt.%) (tr: traces, below 3 wt.%).

	SAG2	SAG10a	SAG10b	SAG11a	SAG12a	SAG12b
Quartz	28	30	33	33	31	32
Feldspars	10	11	12	6	14	13
Illite	11	15	17	13	10	16
Kaolinite	6	6	7	7	5	3
Chlorite	4	6	6	4	4	4
Vermiculite	11	9	4	7	11	9
Calcite	25	18	18	26	22	17
Halite	3	*tr*	*tr*	3	*tr*	3
Pyrite	*tr*	3	*tr*	*tr*	*tr*	3

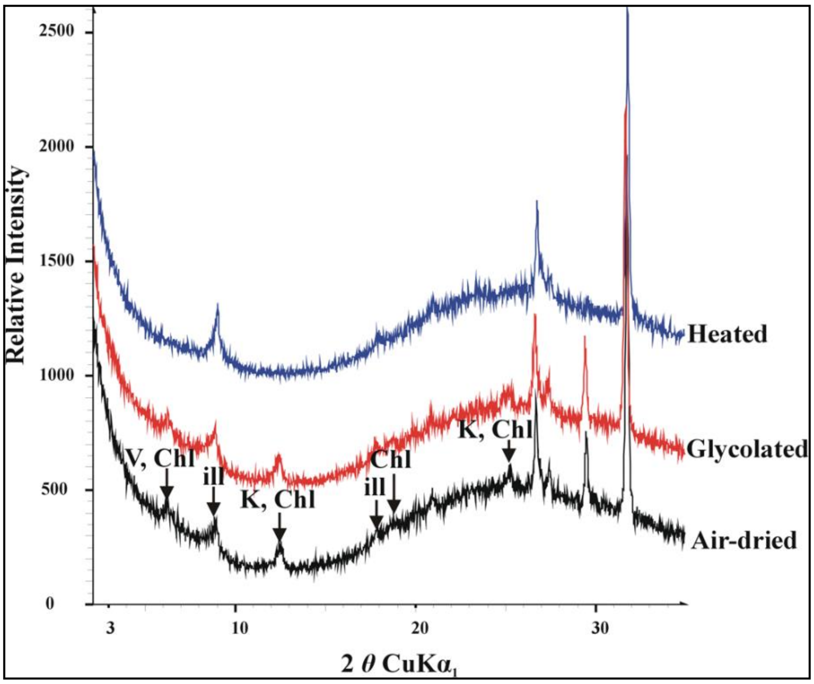

Figure 2 - Clay mineral identification using X-Ray Diffraction (XRD) in the clay fraction of the sample SAG2 (V: Vermiculite, Chl: Chlorite, ill: illite, K: Kaolinite).

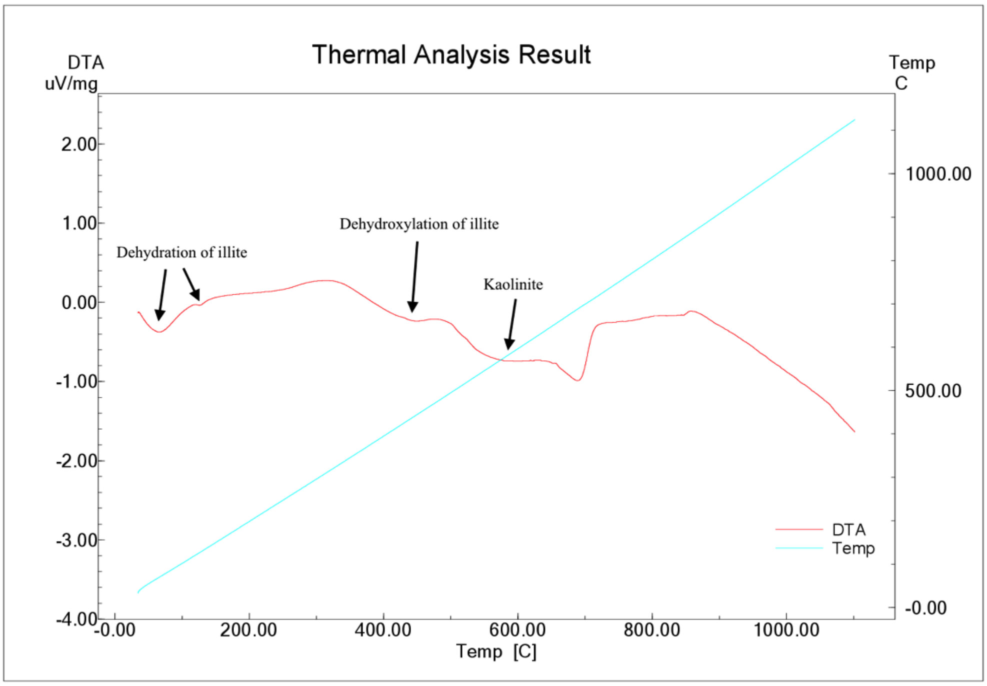

Figure 3 - Differential Thermal Analysis (DTA) of a sample from the Sagiada mud (SAG11).

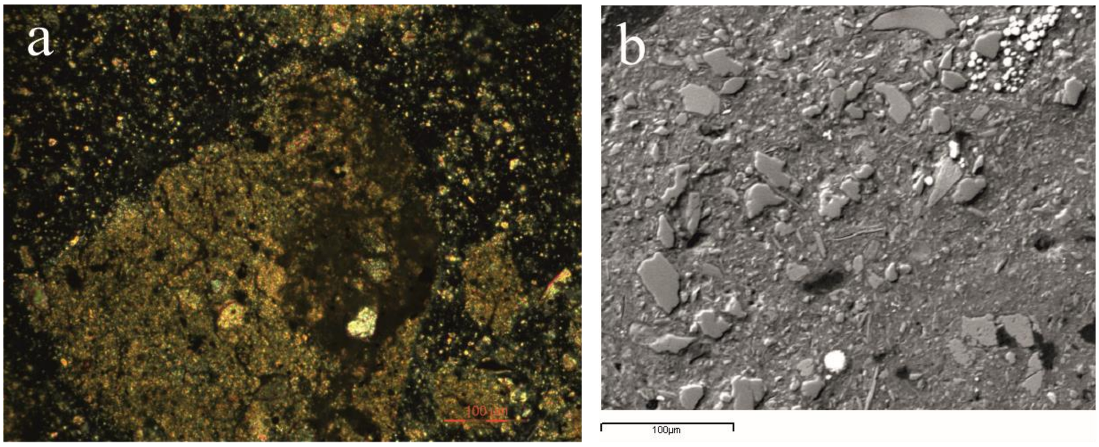

Figure 4 - a) Aggregates of carbonate components and clay minerals from the Sagiada mud, transmitted-light microscopy image, + N, b) SEM image of framboidal pyrite found in the Sagiada mud.

Table 2 - Chemical composition of the Sagiada mud.

	SAG2	SAG10A	SAG10B	SAG11	SAG12A	SAG12B	Mean	Spanish peloids	Floodplain sediments
SiO_2	35.18	43.77	44.61	41.59	40.33	39.67	40.86	18.41 - 51.12	7.90 – 100
TiO_2	0.41	0.55	0.53	0.46	0.47	0.47	0.48	0.22 - 0.55	0.05 – 2.15
Al_2O_3	8.56	10.87	10.64	9.79	9.99	9.73	9.93	3.58 - 12.24	0.10 – 32.6
MnO	0.10	0.13	0.11	0.09	0.10	0.12	0.11	0.02 - 0.61	<0.01 – 6.61
MgO	3.89	5.01	4.64	3.85	3.96	4.20	4.26	0.98 - 22.35	<0.1 – 17.2
CaO	17.39	10.51	10.46	13.42	13.44	14.03	13.21	2.27 - 26.48	<0.05 – 54.4
Na_2O	1.80	1.75	1.78	1.53	1.55	1.59	1.67	0.3 - 4.34	<0.2 – 3.70
K_2O	1.77	2.30	2.21	2.01	2.06	2.02	2.06	0.57 - 2.57	0.11 – 5.10
P_2O_5	0.11	0.12	0.11	0.12	0.11	0.10	0.11	*nr*	<0.01 – 2.61
$Fe_2O_3{}_T$	4.43	6.12	5.65	4.85	4.98	5.06	5.18	1.7 - 5.24	0.25 – 35.8
SO_3^T	1.83	0.93	2.16	1.57	1.81	2.16	1.74	0.06 - 2.37	<0.01 – 1.36
LOI	24.52	17.94	17.1	20.71	21.19	20.86	20.39	13.95 - 53.84	*nr*
As	10	11	11	11	11	19	12	4.4 - 29.6	<5.0 – 410
Ba	160	239	212	182	184	237	202	147.7 - 799	7.00 - 2210
Be	1	2	2	2	3	2	2	*nr*	<0.02 – 47.5
Cd	<1	1	1	1	1	1	1	<10	<0.02 – 23.6
Co	21	26	27	22	24	24	24	4 - 16.8	<1.0 – 55.0
Cr	125	184	195	146	148	163	160	14.6 - 68.2	3.00 - 1600
Cs	<10	<10	<10	<10	<10	<10	<10	<6 - 28.5	<4 – 40.0
Cu	53	50	47	58	54	46	51	11.5 - 52.3	1.00 – 421
Mo	5	2	3	3	3	5	4	<1 - 4.4	<0.05 – 191
Ni	164	194	202	154	157	166	173	3.4 - 50.8	2.00 – 942
Pb	21	22	18	22	21	19	21	10.9 - 37.5	<3.0 – 5200
Sb	0.9	0.9	0.8	1.15	0.9	1.3	1	<2.37 - 4.3	<0.02 – 99.4
Se	<1	<1	<1	<1	<1	<1	<1	<1 - 1.6	*nr*
U	3	2	3	3	3	3	3	<1.20 - 18.4	<1.0 – 89.0
V	93	117	115	118	118	109	112	31.6 - 90.9	3.00 - 140
Zn	207	124	105	122	116	129	134	33.1 - 160.4	7.00 - 2830

Major elements in wt.% and trace elements in mg/kg. Fe_2O_3 and SO_3 refer to total iron and sulphur, respectively. The major element composition is normalized so that the addition of LOI results 100. The compositional range of Spanish peloids is after Carretero *et al.* (2010) and Carretero *et al.* (2014), and the compositional range of European floodplain sediments is after FOREGS (2005). nr: not referred.

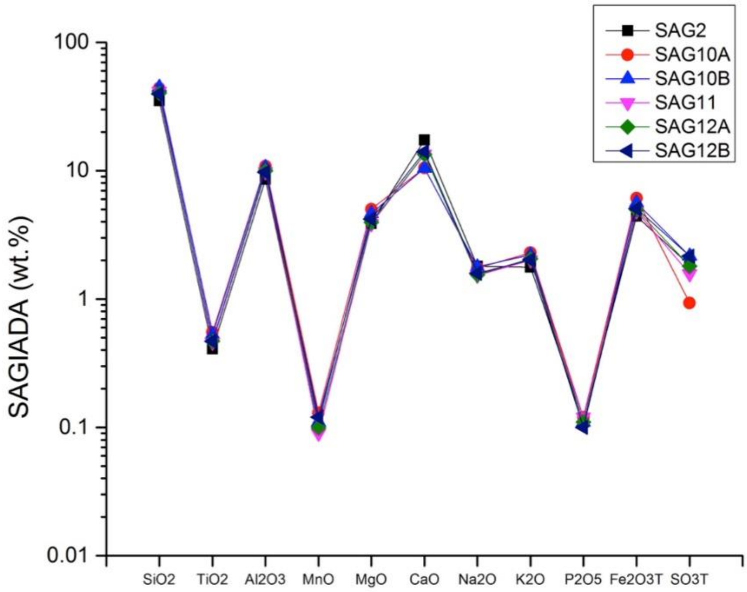

Figure 5 - Major element composition of the Sagiada mud.

5. Concluding Remarks

The Sagiada natural mud occurrences display a significant resemblance in terms of mineralogical and chemical composition. The main mineral phases are quartz, feldspars, illite, kaolinite, chlorite, vermiculite and calcite and minor phases are halite and pyrite. Traces of muscovite and gypsum were also identified. Compared to peloids used in Spanish spas, the Sagiada mud is enriched in Ni, Cr and slightly V and Co, but those concentrations are within the compositional range of floodplain sediments from Europe. The Sagiada mud, compared to the floodplain sediments from Europe has slightly elevated SO_3 concentrations, which are reasonable for therapeutic muds, as indicated by the comparison with Spanish peloids.

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