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MINERAL ABSORBENT EFFIECIENCY ON THE PETROLEUM SPILLS REMOVAL

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ABSTRACT. Mineral absorbents are of great interest for the oil spills removal. Therefore, current investigation tests the removal ability of three commercial products: Zeolit Spectrum, Favisan Clay and professional oil spills removal Adabline II OS. SEM investigation reveals that all compounds relies on small phyllosilicates particles of about 1 – 5 µm accompanied by fewer coarser fractions of 100 – 150 µm. Mineralogical optical microscopy reveals that Zeolit Spectrum and Adabline II OS contains mainly Clinoptilolite while Favisan Clay contains mostly Kaolinite (1 – 10 µm) with some traces of Biotite (5 – 30 µm). These products were tested on diesel and burnt oil spills. The gravimetric measurements reveal the best specific absorption for Clinoptilolite of about 1.26 g/g for diesel and 1.69 g/g for oil while Kaolinite has only 1.04 g/g for Diesel and 1.37 g/g for oil spill. The fact was proved by FTIR spectroscopy revealing the increase of the C=C and C-O. The absorption mechanism was observed by SEM revealing the diesel and oil penetration within the finest mineral clusters, Clinoptilolite being more efficient than Kaolinite which was slightly reluctant because of its hydrophilic nature.

Keywords: *Oil spills, mineral absorbents, zeolite, clays*

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INTRODUCTION

Industrial and transport activities require a lot of fuel like gas, Diesel and kerosene and the vehicle's engines require proper lubrication ensured by specific oils [1, 2]. Both fuels and oils are hydrocarbons products and therefore they are hazardous for environment especially when are spilled on soil or water [3]. The soil contamination affects plants growth and damages the crops which become un-edible [4, 5]. On the other hand, hydrocarbon wastes on the water environment have toxic effect on the aquatic life forms like fish, shellfish and algae [6, 7]. Therefore, absorbent materials are of great interest to be used for the oil spills mitigation. Their nature should be adapted to the environment conditions: the contaminated soils would require a proper granular absorbent able to catch as more as possible the spilled grease and to preserve the soil structure. Water contamination requires absorbent materials with a good floatability that ensures their concentration over the water surface just like the oil spill. Thus, mineral absorbents are most suitable for the remediation of soil contamination while vegetal absorbents are more indicate to be used for decontamination of water surfaces [8].

An absorbent material requires a high specific surface associated with small particles. There is a mineral class fulfilling these requirements which is widespread in the earth crust. This special mineral class is represented by the phyllosilicates which have a lamellar structure based on the silica tetrahedral sheets interlocked by alumina and various alkaline ions such as K, Na and Li. The silica tetrahedral sheets interconnection through intermediary ions forms several subclasses like: Chlorite (e.g. Clinochlore mineral colored in green shades), Micas (e.g. Muscovite, Biotite and Lepidolite) and Clays (e.g. Kaolinite, Montomrillonite and Sericite) [9-11]. Tectosilicates are very similar to the Phylosilicates being structured on the silica tetrahedra bonded each other on their corners forming a light three-dimensional network the most common Tectosilicate subclass are feldspar and zeolites (e.g. Clinoptilolite). Zeolites are known in literature for their permeable structure allowing liquids to penetrate their interplanar spaces and to exchange ions with the adsorbed liquid [12, 13]. It is also mentioned in literature for its oil spills removal abilities [13, 14].

Literature data reveal that phyllosilicates fragmentation occurs through lateral cleavage of the particles followed by their subsequent breaking under the action of external forces forming very small particles of about 1 μm and even having submicron sizes [15, 16]. Such behavior facilitates increasing of the specific surface which is desired for the absorbent properties. Large amounts of kaolinite are found from natural and anthropogenic sources like sedimentary deposits or industrial sludge [17, 18] having a refined micro-structural distribution.

Local sources are very abundant such industrial sources and natural sedimentary deposits like clays deposited in whole Transylvanian basin as consequence of volcanic eruption of Ciomadu Mare Mountain [11, 19, 20]. Clinoptilolite is found in Dej and Racos volcanic tuff deposits within Transylvanian Basin [20, 21]. Therefore, the local deposits can be easily found and used for development of absorbent materials.

The aim of present research is to investigate the absorbent ability of some commercial materials based on clay and zeolite minerals at different exposure like diesel and burnt oil spills using modern investigation techniques such Scanning Electron Microscopy (SEM) and Fourier Infrared Spectroscopy (FTIR).

RESULTS AND DISCUSSION

The materials considered for use as absorbents for oil waste and oil stains have different natures from minerals to vegetal materials and therefore, they have an electrically insulating character. Therefore, these powdery - granular materials were deposited on double-sided carbon tape and investigated by scanning electron microscopy (Scanning electron Microscopy - SEM) in low vacuum mode so that they do not require metallization and the morphological details are perfectly visible.

Zeolit Spectrum sample has a very fine powdery appearance, with most particles having sizes up to 10 μm but there are some fractions larger than about 100 – 150 μm but not very numerous, Figure 1a.

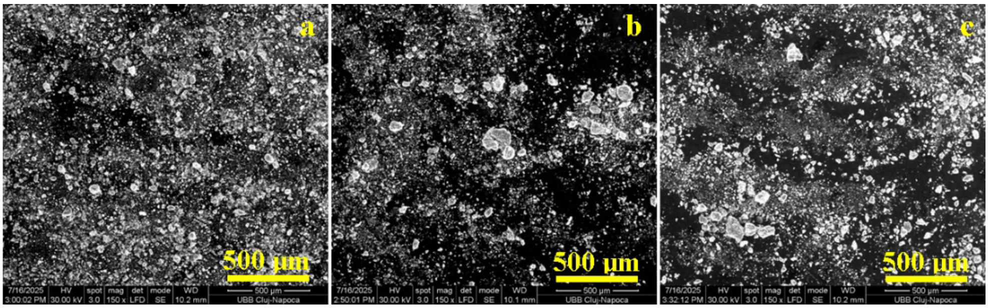

Figure 1. SEM images of the initial mineral absorbents: a) Zeolit Spectrum, b) Favisan Clay and c) Adabline II OS.

Favisan Clay sample is very similar to the previous one having very fine particles predominantly 1 – 3 μm to 10 μm but which tend to agglomerate slowly forming clusters larger than about 50 μm. Some coarse particles

100 – 200 µm with a boulder-like appearance and monolithic texture are also noted indicating an exogenous impurity most likely of a siliceous nature, Figure 1b. The Adabline II OS material has a texture and distribution intermediate between zeolite and clay indicating a mineral component specially conditioned for the absorption of oil and petroleum spills. It has a bentonite look with a high content of montmorillonite, a clay mineral from the smectite category whose crystallographic planes are ideal for liquid absorption, Figure 1c, but the material description indicates the main component as activated Clinoptilolite. It explains the more refined distribution compared to the Zeolit Spectrum's Clinoptilolite featuring coarser fractions.

The targeted absorbing materials have a high content of mineral and/or crystalline material; therefore, each constituent particle must be correlated with the mineral from which it is made. This goal can be achieved by examining the samples with the help of mineralogical optical microscopy that investigates the samples in polarized light with crossed nicols. This method sends a beam of polarized light oriented at 0° that passes through the sample and the analyzer lens (analyzer nicol) is oriented at 90°. Thus, if the sample is missing or if the material is amorphous, then the analyzer nicol does not let any light ray pass and a dark field will be seen in the ocular lens. Crystalline materials cause the plane of oscillation of the polarized light to rotate under a specific angle where colored light maxima will appear in the specific shade of each mineral [18, 22]. Therefore, using this method we can identify the mineral components in the samples to be investigated as well as the dimensional range of the constituent particles, Figure 2.

Zeolit Spectrum sample presents a finely dispersed granular aggregate appearance with an average content of coarse particles with dimensions ranging from 10 to 100 µm as observed in Figure 2a. The microstructural detail in Figure 2b shows that these are surrounded by finer particles with dimensions ranging from 1 to 5 µm. The white-yellowish appearance of the luminous maximum and the greenish-gray extinction are uniform for all particles within the visual field, indicating a high purity of the zeolite. All the observed characteristics indicate its belonging to the Clinoptilolite category according to data from the specialized literature [23, 24]. It has the chemical formula $(Na,K,Ca)_{2\text{-}3}Al_3(Al,Si)_2Si_{13}O_{36}\bullet 12H_2O$ and crystallizes in the monoclinic system. This fact is correlated with the tabular-lamellar appearance of the coarse particles and their angular edges as a consequence of fragmentation by cleavage. Literature data show that the finer the particles in the submicron and nanostructured range, the more capable it is of adsorbing polluting ink stains [24] as well as being effective in desulfurizing crude oil. Therefore, the highlighted microstructure is promising regarding the efficiency of this material in absorbing oil and petroleum stains for the decontamination of solid surfaces.

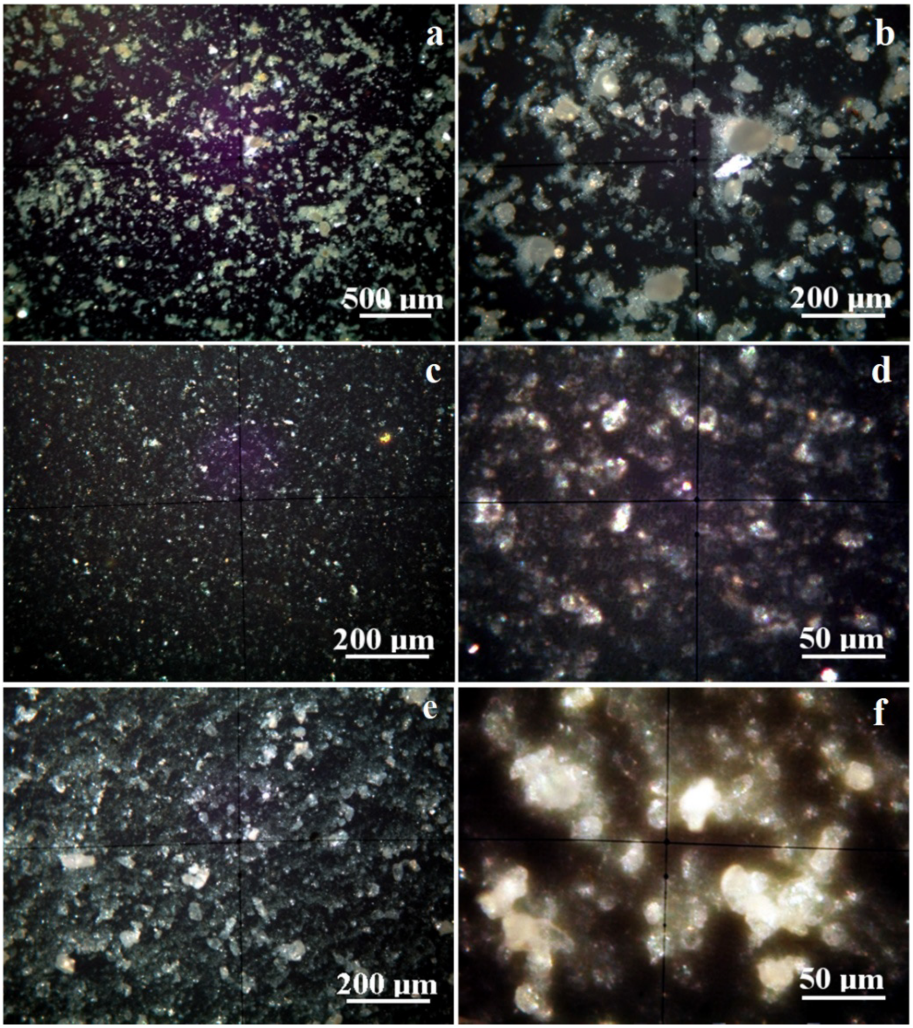

Figure 2. Mineralogical optical microscopy images for the investigated absorbent materials: Zeolit Spectrum a) overall appearance, b) microstructural detail; Favisan Clay c) overall appearance, d) microstructural detail; Adabline II OS e) overall appearance, f) microstructural detail.

Favisan clay is a very fine microcrystalline powder as can be seen in Figure 1c. Most of the particles are extremely fine and have a white hue corresponding to kaolinite which has the chemical formula $Al_2Si_2O_5(OH)_4$ and crystallization in the triclinic system [25, 26]. These are accompanied by some larger fractions having dimensions of about 10 – 50 μm with a tabular lamellar aspect and reddish brown (orange) hue corresponding to biotite, a mineral in the micas category but where certain K atoms are replaced by Mg and Fe having the chemical formula $K(Mg,Fe)_3(AlSi_3O10)(F,OH)_2$ and crystallization in the monoclinic system [27]. Both minerals have foliated aggregates that cleave very fine fractions. This is proven by the microstructural detail in Figure 1d. Here the lamellar-tabular appearance of the kaolinite particles is very clearly observed, most of which are around 1 μm and some are even finer (they could be nanostructured). However, some particles in the range of 1 – 5 μm are very visible accompanied by some finer orange lamellae of around 2 μm corresponding to biotite. Literature data highlight the ability of kaolinite to absorb liquids by penetrating them between the crystallographic planes formed by SiO2 tetrahedra [25, 26]. It is worth noting that natural forest clays may have, in addition to the kaolinitic mass, traces of muscovite and quartz particles [28] but the sample used does not contain quartz traces. Therefore, it is expected that this powdery material will also have a high efficiency in absorbing oil stains.

The Adabline II OS material is currently used for the decontamination of solid surfaces polluted with petroleum and oleic products. Therefore, it constitutes a benchmark for comparison with other granular materials candidates for the decontamination function. The overall microstructural aspect highlights a very fine granular material with a high tendency to cluster. In general, the microstructural clusters in Figure 2e have a dune shape and predominant dimensions around 40 μm. Their hue is whitish, very similar to kaolinite but which could also be a very finely ground zeolite sample. The overall mineralogic observation confirms SEM microstructure. The detail in Figure 2f highlights very fine particles of about 0.5 – 1 μm that have a high tendency to coalescence, generating the microstructural dune – shaped clusters already observed. The fact that no other colored particles appear indicates the single-phase crystalline constitution of the investigated material. Its great similarity to the zeolite and clay considered gives indications that the latter could be largely as effective in removing oil and/or petroleum stains.

Zeolit Spectrum, Favisan Clay and Adabline II OS material have a deep mineral character generated by the tabular lamellar particles specific to phyllosilicates (clays) and tectosilicates (zeolites). These have a special behavior in the case of fragmentation, namely they cleave very easily under tangential stresses and under axial-perpendicular stresses they show high resistance [15, 17]. Therefore, their current state can be improved by mechanical grinding in a ball mill [18, 19], increasing the specific surface area and activating the liquid penetration sites between the crystallographic planes, improving the absorption of petroleum waste.

All investigated samples have great potential for absorbing petroleum spills but a quantitative measurement is necessary. Thus, 100 grams of each sample was tested gravimetrically against diesel and burnt oil spills revealing their specific absorption, Figure 3.

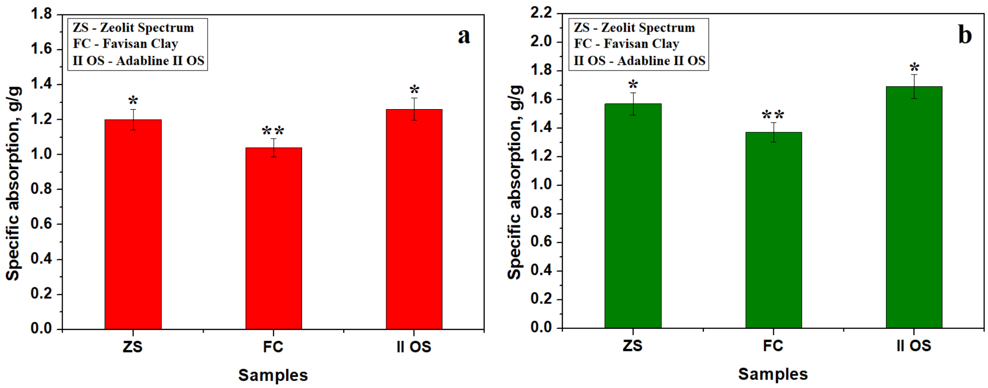

Figure 3. Specific adsorption variation on the mineral samples tested in: a) Diesel and b) Burnt oil.

The specific measured absorption reveals statistical differences between Diesel, Figure 3a, and burnt oil, Figure 3b indicating that the last one is better absorbed by the mineral absorbent comparative to Diesel. The fact is explained by the more viscous nature of oil compared to the significantly volatile behavior of Diesel. Thus, it is expected that the viscous oil adheres better on the absorbent small particles while Diesel favors their relative mobility hindering the formation of removing paste.

There are also found statistical similarities between specific absorption within Zeolite Spectrum and Adabline II OS because of their Clinoptilolite content, thus these samples form the first statistical relevant group. The second statistical group consists in Favisan Clay sample which has a significant lower specific absorption. This behavior is explained by the hydrophilic nature of clays which are prone to absorb aqueous solutions than oily liquids [31, 32]. Avram et.al. evidences the aqueous dispersion ability of the Kaolinite clay withing ceramic slurry being enhanced by the alkaline pH [33]. Wang et.al. reveal that fluoridation of the clay powder switches its hydrophilic behavior into hydrophobic one ideal for oil spills absorption [34]. On the other hand, silanization is also reported to enhance the hydrophobic behavior of the clays [35].

FTIR spectra of mineral absorbents exposed to diesel and burnt oils spills are present in Figure 4 and 5.

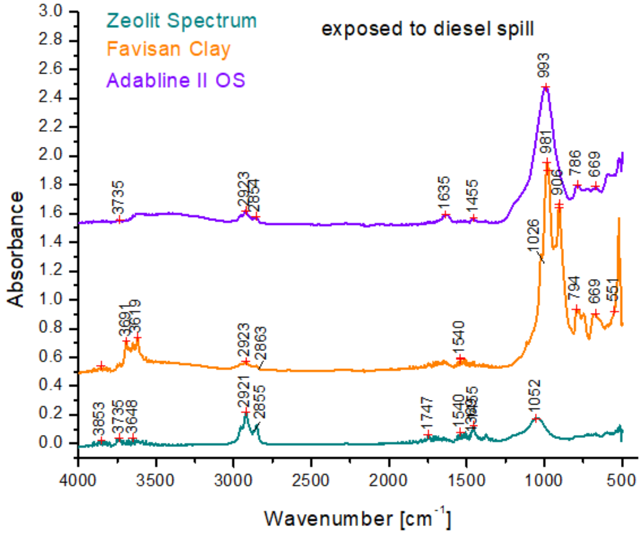

Figure 4. FTIR spectra of mineral absorbents exposed to diesel spill.

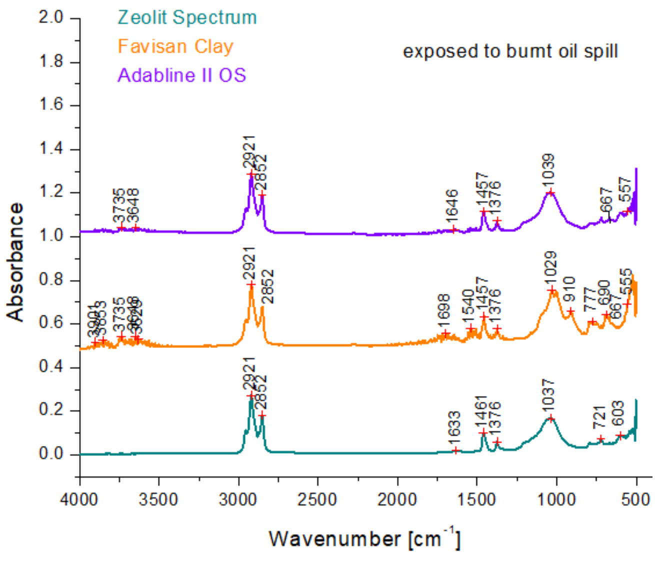

Figure 5. FTIR spectra of mineral absorbents exposed to burnt oil spill.

The absorption bands at 545 cm^{-1} corresponds to O-Si-O bending deformation; 667 cm^{-1} to Si-O symmetrical bending and 1029, 1039 and 1037 cm^{-1} fits the in plane Si-O stretching [36]. These absorption bands are common for all silicates and are induced by the silica thetrahedra. The clay and zeolite presence are particularly evidenced by the absorption band at 777 cm^{-1} belonging to Al-O stretching [36].

The absorption bands at 2921 cm^{-1} and 2852 cm^{-1} corresponds to CH_2 asymmetric and symmetric stretching within both diesel and burnt oil [37]. The C=O chemical bond within diesel is evidenced by the absorption band at 1635 cm^{-1} while the absorption band at 1540 cm^{-1} belongs to C-C stretching within the burnt oil [38].

FTIR spectrum in Figure 4 reveal the previously mentioned absorption bands which are also observed in the spectra obtained on the mineral samples after diesel adsorption. The intensity of CH_2 and C=O absorption bands increase progressively with the spilled liquid absorption. Thus, Favisan Clay reveals lower intensities for the organic bands while their intensity increases progressively within Zeolit Spectrum and the greatest intensities are observed for Adabline II OS. There is observed a significant increase within absorption bands for Si-O stretching and for Al-O stretching indicating Diesel penetration within the crystallographic planes which causes some local reorientation within the phyllosilicate's sheets.

The FTIR spectra in Figure 5 for burnt oil also reveals absorption bands for CH_2 and C=O but also has the characteristic in C-C stretching. The mineral samples after oil absorption are significantly enriched with its specific bands. Their intensity progressively increases with the absorbed amount. Figure 5 reveal that less oil is absorbed within Favisan Clay and much more onto Clinoptilolite based samples because of better properties of zeolite matter. Beside the oil viscosity, the crystal planes are significantly affected by viscous liquid penetration causing mild increase of the mineral absorption bands in the range of 633 – 1033 cm^{-1}.

These morphological changes should influence the absorbents morphology, fact further investigated by SEM, Figure 6.

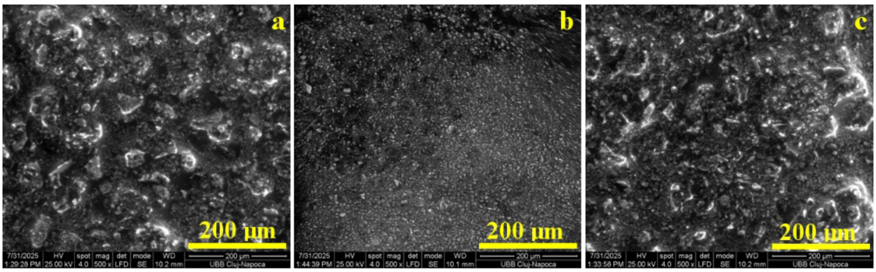

Figure 6. SEM images of the mineral absorbents after burnt oil absorption: a) Zeolit Spectrum, b) Favisan Clay and c) Adabline II OS.

SEM images taken on the absorbent samples after exposure to the burnt oil reveal the spreading of Clinoptilolite particles within Zeolit Spectrum and Adabline II OS within the oil spill absorbing the liquid between the thin crustal sheets, Figures 6a and c, fact in good agreement with FTIR observation regarding enhancing Si-O and Si-O-Si absorption bands intensities. However, the slightly lower absorption of Zeolit Spectrum compared to Adabline II OS is generated by those fewer bigger zeolite particles which do not allow a spread as good as Adabline does. On the other hand, Figure 6b reveals the uneven spreading of the Kaolinite fine particles regarding the oil spill because of the proper modification of their hydrophilicity. It is noteworthy mentioning that the central point of the observation field within Figure 6b and the lower right corner reveal dense areas where clay particles are coalesced through the burnt oil justifying the measured specific absorption.

The physicochemical aspects reveal that the local product Adabline II OS has the best behavior in removing both diesel and burnt oil spills. Zeolit Spectrum is a purified Clinoptilolite destined for human consumption as food supplement and therefore it is very expensive to be used in large quantities for petroleum spills removal. Fortunately, Transilvanian Basin is very rich in volcanic tuff containing both clays (Clinochlore, Muscovite and Kaolinite) but also Clinoptilolite zeolite and can be used for petroleum spill removal with minimal conditioning [39, 40].

Favisan Clay is a pure Kaolinite clay destined for cosmetic usage and therefore it is too expensive for be used for petroleum stains removal. It was used to assess the pure Kaolinite ability in oil spill removal. The lack of fluoridation or silanization treatments to increase its hydrophobicity makes it inferior to the zeolite-based materials. But there are large local clay deposits containing various proportions of Kaolinite and Muscovite which can be easily exploited in Transylvanian Basin generating cost effective oil spill removal agent with good behavior. It is strongly recommended that industrial agents working with large amount of petroleum to have a moderate stock of clay-based absorbent to be used in case of stringent necessity.

The mineral absorbent has a major lack regarding their neutralization after use because they cannot be completely destroyed by burning. They should be deposited in controlled dumps to avoid re-spilling of the absorbed hydrocarbons. The future challenge is how to recover the used mineral absorbents through the progressive filtration or development of a composite byproduct.

This study focused on high-purity, locally sourced materials to evaluate the specific absorption behavior of clinoptilolite and kaolinite with respect to diesel and burnt engine oil. The high cost of products like Zeolit Spectrum or Favisan clay poses a significant limitation to their use as petroleum absorbents.

In contrast, Adabline II OS is a purpose-designed product well-suited for this application. Consequently, a key direction for future research is the in-depth investigation of low-cost local clays, aiming to determine the optimal conditioning methods (such as grinding and/or silanization) needed to achieve effective oil stain absorption.

CONCLUSIONS

The tested local mineral products are effective on the diesel and burnt oil spills removal. The best results are obtained by the Clinoptilolite based materials (Zeolit Spectrum and Adabline II OS) because of their optimal micro porosity and absorption availability generated by the crystal structure. Adabline II OS prove to be very balanced product acting optimally for quick removal of the petroleum spills, while Zeolite Spectrum is not a specific destined product.

Kaolinite sample has slightly lower specific absorption because of its natural hydrophilicity. It requires special treatments to increase its hydrophobicity like silanization. But it can be used as emergency material to mitigate the petroleum spill if the specialized product is missing.

Natural deposit of volcanic tuff within Transylvanian Basin could be a valuable source for production of a cost-effective mixed zeolite/clay mixture to be used for mild spills removal.

EXPERIMENTAL SECTION

The commercial absorbent materials were purchased directly from their supplier as follows: Zeolit Spectrum (Novo Biomedics, Constanta, Romania); Favisan Clay (Favisan, Lugoj, Romania) and Adabline II OS (Adabline, Bucharest, Romania).

The samples morphology in initial state and after absorption was investigated by Scanning Electron Microscopy (SEM) using an Inspect S (FEI Company, Hillsboro, OR, USA) microscope operated in the low vacuum mode at an acceleration voltage of 30 kV.

The crystalline nature of the investigated samples was investigated through the Mineralogical Optical Microscopy (MOM) operated in cross polarized light on a Laboval 2 microscope (Carl Zeiss, Oberkochen, Germany). Each powder was carefully spread on the glass slide allowing its optimal view. The microscopy images were digitally acquired through a Samsung 10 MPx photographic system.

The quantitative absorption experiment was effectuated by weighing 100 grams of each powder samples which was spread on the contaminated surface. They were let to act for 30 minutes and afterward collected and weighed. Reporting the weight after decontamination to the initial absorbent weight results the specific absorption expressed in gram of petroleum pollutant per gram of absorbent used. The experiment was carried out in triplicate and the standard deviation is displayed as error bars in the variation graphs. The values were statistically analyzed using Anova test followed by Tukey post hoc at a significance level of 0.05. The statistical analysis was performed with Microcal Origin Lab version 2018b software (Microcal Company, Northampton, MA, USA).

Fourier Transformed Infrared Spectroscopy (FTIR) was effectuated with JASCO 610 spectrophotometer (JASCO International Co., Ltd., Tokyo, Japan) in ATR method with a resolution of 4 cm^{-1} and 100 scans for each spectrum.

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