

Preparation of Chromatographic Media from Algerian Diatomite

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Abstract

X-ray photoelectron spectrometry combined with FTIR spectroscopy, X-ray diffraction analysis (XRD), and scanning electron microscopy (SEM) allowed highlighting the different interactions of grafts or impregnated polymers on the mineral surface of diatomite obtained by treatment of raw diatomite under reflux with a 5 M solution of hydrochloric acid for 24 h. This acid treatment led to almost total elimination of organic impurities with a total decalcification of the diatomite, leading to its deferrization and partial dealumination, and the obtaining of a support with a homogeneous siliceous surface mainly constituted of Si–OH and Si–O–Si functional groups. The surface of the diatomite washed with HCl became totally saturated with PEG-20M and SE30 with respective impregnation rates of 5 and 2 %. However, the grafting and/or impregnation of this diatomite with octadecyltrichlorosilane (C18) and docosane (C22) led to the formation of perfectly homogeneous surfaces.

Keywords

Diatomite, characterization, grafting, impregnation, x-ray photoelectron spectroscopy (XPS), surface properties

1 Introduction

Knowledge of the surface characteristics of solids, such as the nature and distribution of chemical groups, mineral or organic impurities, surface energy, and morphology is necessary to understand and prevent their behaviour, in particular, if they are in contact with gases, liquids or other environments such as polymers. Among the reinforcing fillers used in industry, a large part is reserved for synthetic silicas.^{1,2} Although these fillers have interesting reinforcing characteristics, economically the manufacturing cost is estimated to be high.^{1,2} Therefore, it makes sense to use natural silicas such as diatomites (diatomaceous earth or Kieselghurs). Natural silica is both abundant in nature and ecological,^{3,4} but it is less pure.¹ In fact, the use of diatomaceous earth as a filler is one of the many possible uses. More specifically, diatomites are the basis of gas or liquid chromatographic media (HPLC).⁵

Furthermore, noted should be the use of these products in the production of drinking water as a clarifying agent, as a powerful filtering agent, catalyst support, abrasive agent, anti-explosive agent, in the manufacture of refractory bricks used in thermal insulation, etc.^{1,4,6–8} For all these applications, the size, morphology, nature, and quantity of mineral impurities and obviously the surface properties are the factors that play a determining role. These examples show that mastering the use of diatomites requires better knowledge of their surface properties. This was the fundamental goal of this study. The XPS provides information on the chemical composition of a surface layer, leading to

the physicochemical characterization of surfaces and interfaces.^{6,7} It is a very well established technique in the field of science and technology of different materials. Furthermore, to our knowledge, there are not many XPS studies in the literature concerning the characterization of polymers deposited on chromatographic support, using Algerian clays^{9,10} and diatomite of Sig.⁵ In addition, no XPS studies of alkyl-grafted diatomite and long-chain normal kerosene grafted/impregnated diatomite on chromatographic support have been performed.

This study therefore continues the previous work entitled “Characterization of an Algerian diatomite by inverse gas chromatography: Specific and non-specific contribution and Lewis acid base parameters”. The aim was to be able to better exploit diatomite for commercial purposes. Indeed, understanding the behaviour of a solid is linked to understanding the interactions on its surface. In this sense, XPS, FTIR, XRD, SEM, and IGC-DI techniques (previously published article) made it possible to monitor the modifications of the surface properties of the various powders examined as a function of the treatment method used.

2 Experimental

2.1 Materials

The diatomite, used in this study as a mineral filler for the different materials studied, was extracted from a deposit in the Sig region of western Algeria. The solvents used for the impregnation were dichloromethane (Aldrich 99 %) and toluene (Prolabonorma pur min 99.99 %). The polar and

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non-polar stationary phases were, respectively, polyethylene glycol (PEG-20M), polydimethyl siloxane (SE30), as well as amino acid (glycine used for the double impregnation). In addition, octadecyltrichlorosilane (C18) was used for grafting, and long-chain alkyl, and docosane (C22), for impregnation of the grafted diatomaceous earth. All compounds were of high analytical quality.

2.2 Preparation of the diatomite and the various supports

The crude diatomite was treated under reflux with a 5 M hydrochloric acid solution for 24 h. The obtained diatomite was washed several times with distilled water until a negative test was obtained with silver nitrate solution of the wash water, and then dried in an oven at 120 °C for 24 h.

Impregnation of diatomite treated with PEG-20M in 50 ml dichloromethane and SE30 in 50 ml toluene was achieved with 2 and 5 %.^{11–13} A double impregnation of the prepared diatomite was obtained by simultaneous 2 % glycine and 5 % PEG-20M. The grafted diatomite was obtained by chemical grafting using the method of Kovats and reported by *Al-Saigh et al.*¹³ on HCl treated diatomite. This graft was then impregnated with a long-chain alkyl, docosan.

2.3 Characterization of the different chromatographic supports

In order to characterize the surface of the different chromatographic supports, X-ray photoelectron spectroscopy (XPS) was used, complemented by X-ray diffraction (XRD) analysis and Fourier transform infrared (FTIR) spectroscopy, to explain the inorganic and organic source of the carbon atoms identified in the XPS spectrum of the crude diatomite (DB). The XRD spectrum showed, through the peaks present, the mineral nature of the carbon structure of the DB in the form of calcium carbonates, such as calcite, dolomite, and H-magaiite, which disappeared upon acid treatment. These techniques made it possible to acquire the information necessary for the study of both the morphology of surfaces and their chemical compositions, but also to establish possible correlations between the different preparation methods and the specific and non-specific properties of the surface of the supports.

The XPS apparatus is a VG XPS LAB MKI spectrometer with radiation from the source of Mg K α , Al K α , with a power of 200 W (20 mA, 10 kV). The latter was applied at the source, with a high energy equal to 20 eV.

The XRD analysis was determined by a PW-1710 BASED type diffractometer, comprising a high voltage generator of 40 kV and intensity 30 mA, with a copper anticathode of length Cu of 1.5406 Å. Infrared spectra were obtained on a Fourier transform Magma IR spectrometer, using a Michelson interferometer over the range of 4000–380 cm⁻¹. The various chromatographic supports were previously encapsulated in KBr pellets. The surface morphology of the different supports was examined by scanning electron microscopy (SEM).

3 Results and discussion

3.1 Surface properties of diatomaceous earth powders: DB and DH

Fig. S1 represents the wide scan XPS spectra obtained for the DB and DH support, respectively. It appears that the main surface elements detected are silicon, oxygen, carbon, and aluminium in trace amounts. The DH spectrum shows an intense peak in oxygen centred at about 530 eV, a main Si2P line coming from silica, as well as a Si2S line. A minority peak centred at about 285 eV was also observed due to carbon contamination of the surface. This was inevitable under the operating conditions of a residual pressure of $5 \cdot 10^{-8}$ mbar.

Fig. 1 illustrates the elementary surface compositions of DB and DH powders, given in atomic percentages of the main elements. Here, a similar elemental composition for the two powders was also noticed.

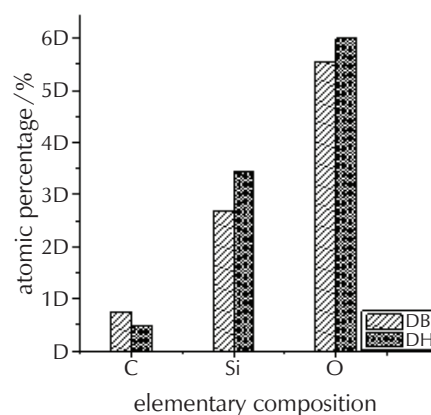


Fig. 1 – Elementary composition of DB and DH surface

In addition, observed was a significant carbon content on the surface of the DB. On the other hand, the percentages of Si and O increased for DH, while that of C decreased. In fact, the DH was washed away from the impurities present on the surface of the DB. This was confirmed by the values of the elemental analysis, recorded in Table 1.

Table 1 – Elementary composition of DB and DH surface

Elements	C	O	Si	Na	Al _{2p}	Fe	Mg _{2p}	Ca _{2p}
DB	7.5	55.5	26.8	1.2	2.6	$1.3 \cdot 10^{-3}$	5.4	0.9
DH	4.9	59.9	34.4	0.0	0.4	0.0	0.0	0.0

Fig. S2 shows the high-resolution XPS spectra of the C1s signal obtained for the two materials examined (DB and DH). The DB spectrum clearly showed a complex structure with an apparent peak at 285 eV with three shoulders at 284, 287, and 290 eV. Their responsibilities are as follows:

- 284 eV: C_{sp2}
 285 eV: C–C, C–H, C–Si
 287 eV: C–O
 290 eV: $>C=O$ (CO_3)

The main peak was attributed to carbon atoms from organic aliphatic groups. Moreover, one can note a significant carbon content at the surface of the DB due to the presence of carbonates also demonstrated by the XRD and FTIR spectra, presented in Figs. 2 and 3, respectively. The XRD spectrum of DH showed strong reflections from quartz. As for those of calcium carbonates in the form of calcite, dolomite, and H-magaiite, they were absent because the reflux treatment of the raw diatomite with a 5 M hydrochloric acid solution resulted in the elimination of the crystalline phases of calcite, dolomite, magaiite, and the retention of the single crystalline phase of quartz. This led to an increase in the amorphous phase and a decrease in the crystalline phases (amorphization).

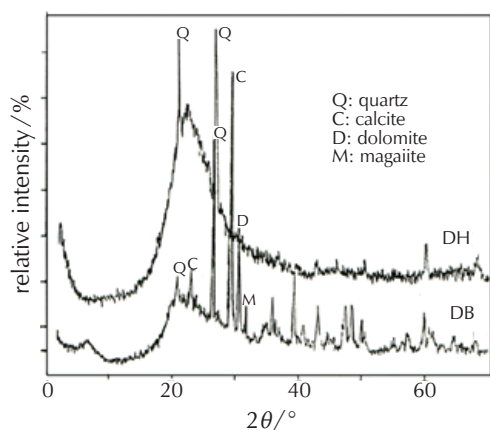


Fig. 2 – XRD spectrum for DB and DH

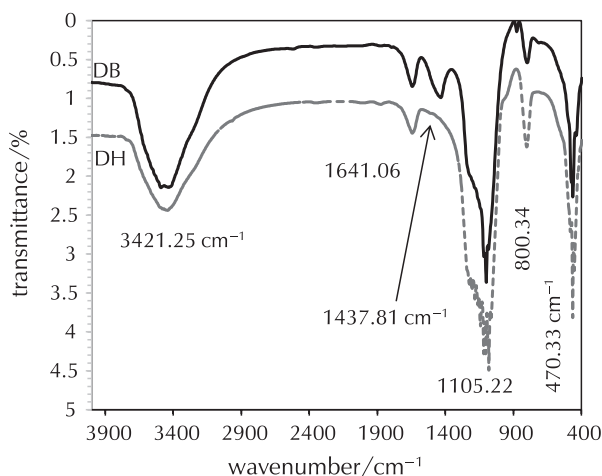


Fig. 3 – FTIR spectrum for DB (—) and DH (---)

The spectroscopic study (FTIR) compared the two spectra before and after washing with 5 M HCl acid at 100 °C. It

was noted that the absorption bands corresponding to the vibrations of CO_3^{2-} were absent from the spectrum of DH. Only the SiO_2 vibration bands were visible.

The surface organic contamination of DH demonstrated by XPS, FTIR, and XRD was low compared to that of DB, since washing with 5 M hydrochloric acid under hot reflux removed the majority of the weakly adsorbed and surface-coating organic contamination as carbon due to organic contamination was identified by the peak centred at 285 eV (C–C, C–H, C–Si), while carbon from dolomite and calcite was identified by the peaks centred at 287 eV (C–O) and 290 eV ($>C=O$ for CO_3^{2-}). After treatment with 5 M HCl acid under reflux for 24 h, this material showed a significant decrease in surface tension due to the pronounced polar character of HCl enhanced by heat. However, the value of the dispersive contribution γ_s^d at 90 °C remained high ($\gamma_s^d = 135.82 \text{ mJ m}^{-2}$) compared to the DB at 140 °C ($\gamma_s^d = 165.01 \text{ mJ m}^{-2}$)¹⁵ generated by the increase of Lewis acid-base interactions (dispersive contribution) caused by the acid.

The latter suggests that DB is a material of high surface energy, with a varied structure. The results obtained are in agreement with those of the literature for a crystalline silica.^{16,17}

Microporous materials have high energy sites inside pores.^{18,19} Such sites can irreversibly adsorb molecular species if they have not been fully activated beforehand. In addition, these porous materials have a regular cylindrical structure, consisting of original pore patterns for DB and DH, as seen in Fig. 4. Similar observations on the morphology of Algerian Sig diatomite have been reported by several authors.^{3,5,21,22}

In addition, there is a significant influence of washing on the acid-base behaviour of diatomaceous earth powders. In fact, washing leads to an overall and significant increase in the values of ΔC^{AB} .¹⁵ This corresponds to an accentuation of the acidic character and the basic character of the

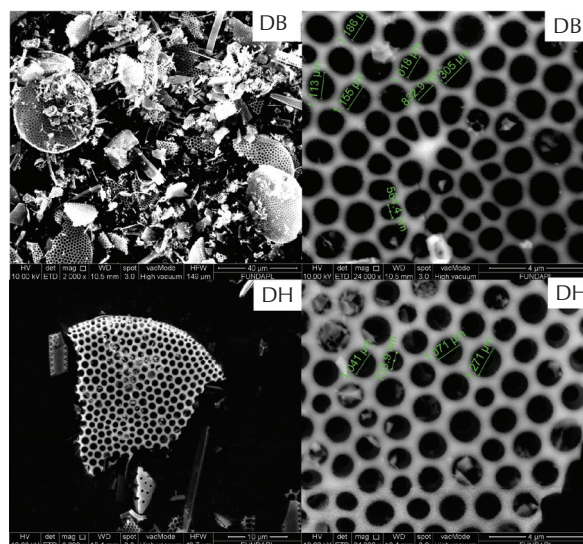


Fig. 4 – SEM images of DB and DH

surface. This result is consistent with the hypothesis that washing with hydrochloric acid partially removes organic pollution by hydrolysis but does not have the oxidizing power of nitric acid against refractory organic compounds, and therefore activates the mineral surface through its release of organic impurities and its acidification.^{5,22}

3.2 Surface properties of diatomaceous earth powders: DHP, DHS, and DHC

The surface chemical composition of DHP, DHS, DHC materials shown in Fig. 5 as obtained from XPS spectra, were intentionally limited to the main elements detected. These elements were comparable to those previously found on the surface of DB and DH. The nature of the elements found on the surface was also consistent with the theoretical formulas of mass compounds.

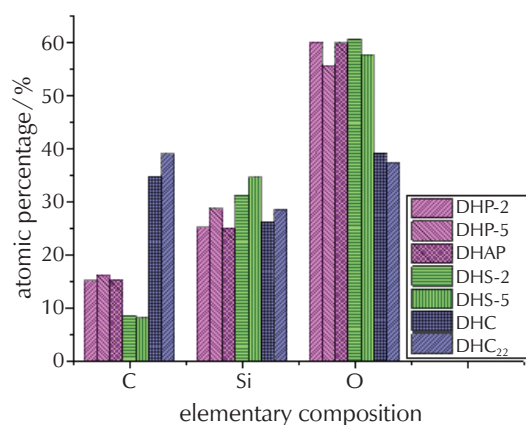


Fig. 5 – Elemental surface composition of DHP, DHAP, DHS, and DHC

As might be expected, the carbon content increased with the amount of organic material deposited on the surface of the samples. In addition, a decrease in the oxygen and silicon content was observed, which was attributed to the masking of the elements of the mineral surface by the organic deposit. However, the silicon level remained constant for DHS, probably due to impregnation with SE30, a polymer partly made of silica.

On the other hand, the SEM images, given in Fig. 6, showed that whatever the sample, the nature of the polymer or grafting, whether it had undergone a hydrophobic chemical modification or impregnation, the appearance of the support remained the same after deposition. The images of the different deposits revealed an almost identical structure with a very porous surface. It also appeared that the entire surface of the support was covered.

Moreover, according to the SEM image of the DHP, it was observed that the pore diameters were between 1 and 3 μm . As for the shape, it was spherical. The polydispersity in particle size for all samples examined was also observed.

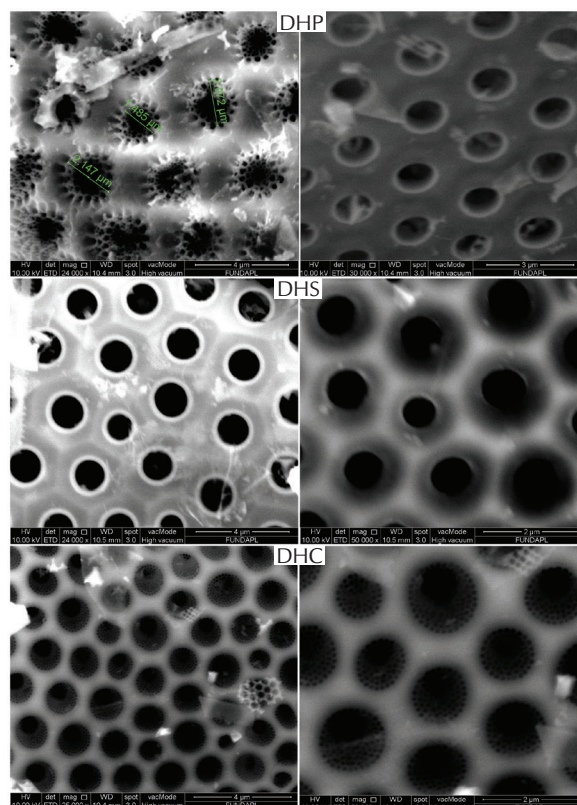


Fig. 6 – SEM images of DHP, DHS, and DHC

3.3 Study of the interactions between the diatomaceous earth support and the polymer and/or graft

Fig. 7 represents the elemental composition of the surface in atomic percentage of the bare material (DH) and of the materials DHP, DHS, and DHC. According to the latter, the masking of elements on the mineral surface was well distinguished. This reflected good wettability (adhesion).²² As for DHAP, which was a diatomite impregnated with an amphoteric amino acid of pH 5.9, the ΔC^{AB} specific interactions were almost identical with those of DHP.¹⁵ However, weak interactions with the THF and dioxane (basic probes) were noted compared to the material DHP-5 at 90 °C. This was also confirmed with chloroform (acid probe), where there was a slight increase in the ΔC^{AB} of DHAP. This suggested that the introduction of glycine (2 %) into the material had not affected the acid-basic character (the basicity of polyethylene glycol prevails). Similarly, for the dispersive component, the values of γ_s^d at 45 and 90 °C remained close to those of DHP-5, but with a slight decrease reflecting better adhesion. With regard to DHP and DHS impregnated powders, the following may be noted:

Fig. S2 shows the presence of a main peak at 286.5 eV, thus confirming the attachment of the polymer to the surface of the DH with a C–CO bond. This adhesion can also be interpreted by the results of the IGC-DI determined by the dispersive component γ_s^d .

A significant decrease in γ_s^d was observed as a function of the mass content of the polymer at the surface of the samples. The result clearly showed that the polymer wetted the

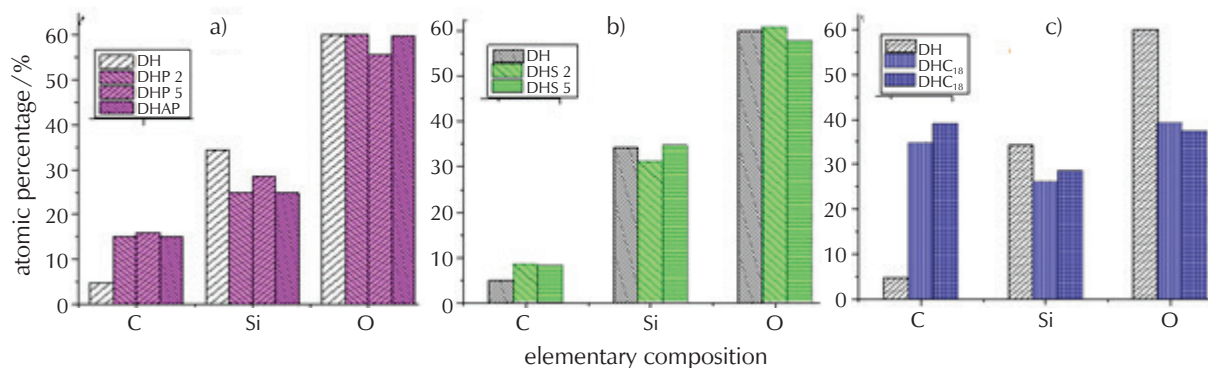


Fig. 7 – Comparison of the elementary composition of the surface of a) DHP, b) DHS, and c) DHP with that of DH

surface of the diatomaceous earth, and that the coverage of the mineral surface increased with the amount of organic deposit, with the exception of DHP at 90 °C.¹⁵

The surface of DHP and DHS powders appeared to be saturated from 5 and 2 wt % at 90 °C, respectively. Indeed, Fig. 8, illustrating γ_s^d as a function of the recovery rate, shows the appearance of a plateau, γ_s^d . At saturation, the addition organic matter no longer improved the recovery of the mineral surface.²²

This result showed that, at saturation, the surface of these diatomite powders was uniformly covered by the polymer. This was confirmed with the SEM images presented in Fig. 6. Finally, the acid-base properties of the coated powders showed that ΔG^{AB} decreased as a function of the content of impregnated organic matter, except for DHP. Regarding the powders grafted and/or impregnated with docosane, their acid-base properties varied uniformly, and exhibited an amphoteric character with a more marked acidity.¹⁵

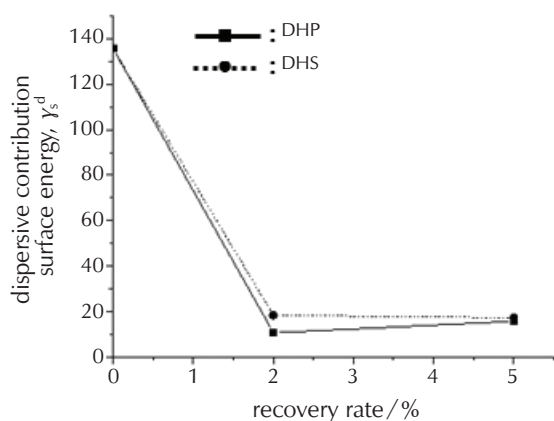


Fig. 8 – Dispersive contribution to surface energy, γ_s^d , depending on the recovery rate

4 Conclusion

X-ray photoelectron spectroscopy (XPS) was used to study the surface properties of bare and coated diatomaceous earth powders. From the results obtained with different analysis techniques, it was observed that the intense treatment with HCl caused total decalcification of the diatomaceous earth, leading to its deferrization and its partial dealumination. The surface obtained had chemical homogeneity and a low specific surface ($5 \text{ m}^2 \text{ g}^{-1}$). This diatomaceous earth powder had a siliceous surface, characterized by a surface functionality of the Si–OH, Si–O–Si type, as well as traces of Al_2O_3 .

On the other hand, washing led to a decrease in part of the organic pollution, and thus activated the mineral surface. However, the surface contamination demonstrated by XPS, FTIR, XRD, and SEM seemed not affect the surface of the DH. However, the level of impurities remained negligible. The masking of the elements on the mineral surface revealed by XPS, represented by the elemental composition of the surface in atomic percentage of the bare and coated diatomaceous earth, confirmed this adhesion.

In addition, the attachment of polymers PEG-20M and SE30 to the surface of DH was quite distinct for both materials, DHP and DHS, represented by the high resolution spectrum of C1S. The surface of these two DHP, DHS powders appeared to be saturated from 5 and 2 % by mass at 90 °C, respectively. The powders grafted and/or impregnated with docosane reflected a more homogeneous surface when passing from DHC18 to DHC22. In addition, this technique (XPS) made it possible to reveal the interactions of grafts or polymers on the mineral surface diatomite. The collection of all this information, by the various techniques used, led to a better development of these materials based on diatomite, with a view to obtaining stationary phases in chromatography.

ACKNOWLEDGEMENTS

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List of abbreviations and symbols

Abbreviations and descriptions of the different diatomite-based media, washed, impregnated and/or grafted

Abbrev.	Description of materials	Column temperature/°C
DB	Raw diatomite	140, 170
DH	Diatomite washed with hydrochloric acid 5 M	90, 140, 170
DHP	Diatomite washed and impregnated with PEG-20M	–
DHP-2	Diatomite washed and impregnated with 2 % PEG-20M	45, 90
DHP-5	Diatomite washed and impregnated with 5 % PEG-20M	45, 90
DHAP	Diatomite washed and impregnated with 2 % Glycine and 5 % PEG-20M	45, 90
DHS	Diatomite washed and impregnated with SE30	–
DHS-2	Diatomite washed and impregnated with 2 % SE30	45, 90
DHS-5	Diatomite washed and impregnated with 5 % SE30	45, 90
DHC	Diatomite grafted with long-chain alkyl	–
DHC ₁₈	Diatomite grafted with octadecyl	45, 90
DHC ₂₂	Grafted diatomite, impregnated with 2 % docosane	45, 90

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SUPPLEMENTARY MATERIALS

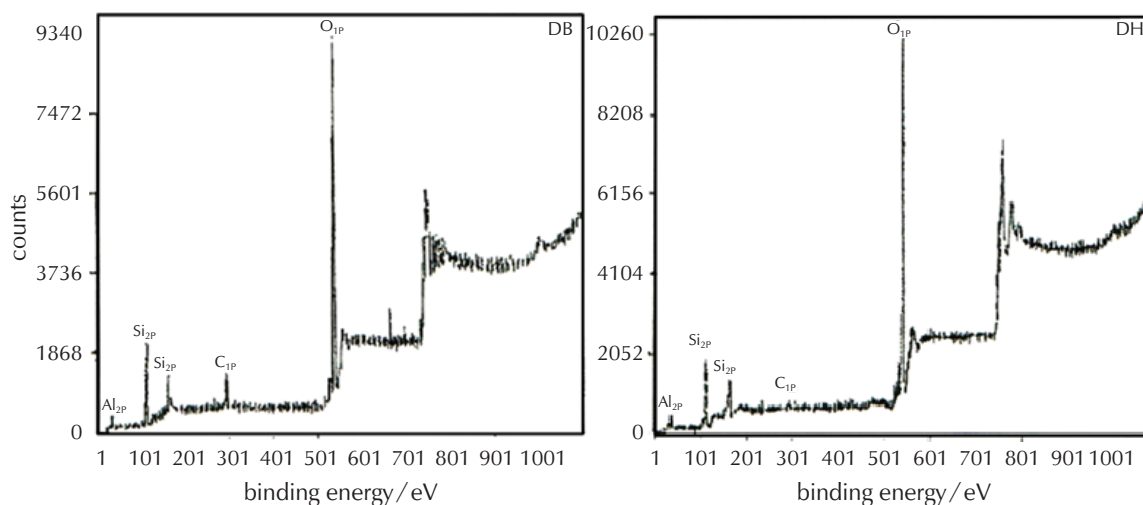


Fig. S1 – Wide scan XPS spectrum of DB and DH

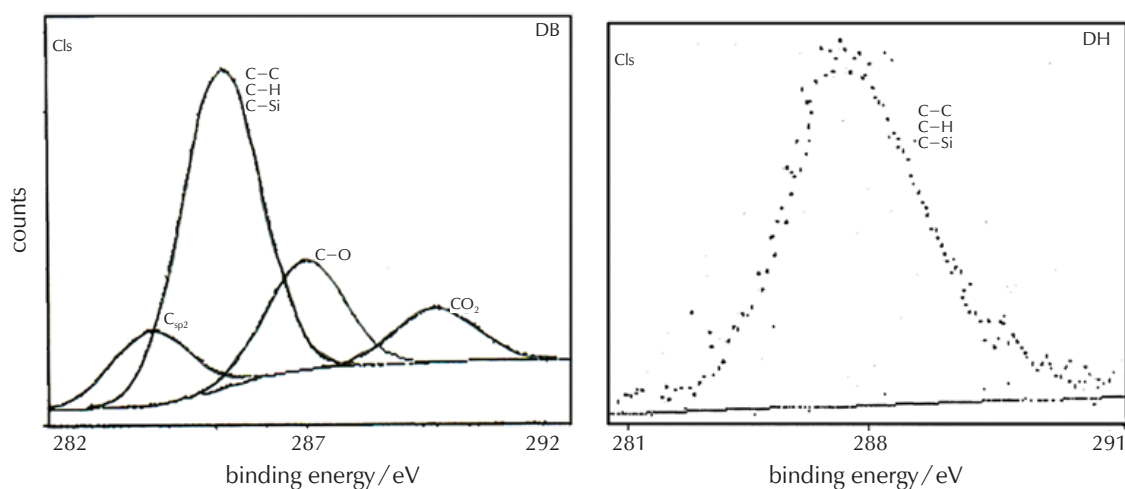


Fig. S2 – High resolution XPS spectrum of C_{1s} for DB and DH

SAŽETAK

Priprema kromatografskog medija iz alžirskog dijatomita

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Rendgenska fotoelektronska spektroskopija u kombinaciji s FTIR spektroskopijom, rendgenskom difrakcijskom analizom (XRD) i skenirajućom elektronskom mikroskopijom (SEM) omogućila je uvid u različite interakcije graftova ili impregniranih polimera na mineralnoj površini dijatomita dobivenih obradom sirova dijatomita pod refluksom 5 M otopine klorovodične kiseline tijekom 24 h. Navedena kiselinska obrada gotovo je u potpunosti uklonila organske nečistoće uz potpunu dekalifikaciju dijatomita, deferizaciju i djelomičnu dealuminaciju, uz dobivanje kvarcne površine koja se uglavnom sastojala od funkcionalnih skupina Si–OH i Si–O–Si. Površina dijatomita isprana HCl-om u postala je u potpunosti zasićena PEG-20M-om i SE30-icom s impregnacijom od 5 odnosno 2 %. Graftiranje i/ili impregnacija dijatomita oktadeciltriklorosilanom (C18) i dokožanom (C22) dovela je do stvaranja savršeno homogenih površina.

Ključne riječi

Dijatomit, karakterizacija, graftiranje, impregnacija, rendgenska fotoelektronska spektroskopija (XPS), svojstva površine

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