Bulgarian Chemical Communications, Volume 47, Special issue C (pp. 79–86) 2015

Preparation of Fe-Pd/Al-Si-O catalyst using biogenic iron of cultivated *Leptothrix* genus bacteria

M. G. Shopska[1]*, G. B. Kadinov[1], J. Briančin[2], I. D. Yordanova[1], H. G. Kolev[1], M. Fabián[2]

[1] *Institute of Catalysis, Bulgarian Academy of Sciences, Acad. G. Bonchev St., Bldg. 11, 1113 Sofia, Bulgaria*
[2] *Institute of Geotechnics, Slovak Academy of Sciences, 45 Watsonova St., 040 01 Košice, Slovakia*

Received: September 15, 2015;   Revised November 4, 2015

A composite has been prepared by modification of 0.3%Pd/Al-Si-O fibrous catalyst, synthesized by a sol-gel method, with a biogenic material upon applying cultivation of *Leptothrix* genus of bacteria in a silicon-iron-glucose-peptone elective medium. Samples were characterized by infrared spectroscopy, scanning electron microscopy with energy dispersive X-ray analysis, low temperature adsorption of nitrogen, and X-ray photoelectron spectroscopy. The hybrid material contained biogenic iron γ-FeOOH. Catalytic activity in the CO oxidation reaction was monitored by *in situ* diffuse-reflectance infrared spectroscopy. Catalytic activity tests were aimed at evaluating the catalytic performance and examining a possibility to prepare iron-containing catalysts with biogenic material. Our study showed similarities between catalytic performances of the studied systems, which are assigned to uniform and stable distribution of the palladium and iron on the surface. The catalytic activity of synthesized composite was essentially due to Pd.

Keywords: biogenic iron-containing material; *Leptothrix* genus of bacteria; X-ray photoelectron spectroscopy; CO oxidation; *in situ* diffuse-reflectance infrared spectroscopy; scanning electron microscopy with electron dispersive analysis.

INTRODUCTION

Biogenic iron-containing materials are applicable in the heterogeneous catalysis in gaseous or liquid medium playing different roles as a catalyst precursor, active component, catalyst support, and immobilizing carrier. Investigations are concentrated on reactions of oxidation, degradation of dyes, transformation of secondary alcohols, formation of intermediates for pharmacy and organic synthesis, CH_4 cracking, mediation in dechlorination of Cl-containing hydrocarbons [1–11].

Studies of natural biogenic material, a product of *Sphaerotilus-Leptothrix* group bacteria metabolism collected from small water streams at Vitosha Mountain (Bulgaria), showed that an iron-containing ingredient is goethite [3]. Biogenic iron-containing materials obtained by laboratory cultivation of *Leptothrix* genus of bacteria in different growth media contained mixtures of α-FeOOH, γ-FeOOH, and γ-Fe_2O_3 [12]. Both the natural and laboratory obtained biogenic materials exhibited some catalytic activity in the reaction of carbon monoxide oxidation [3, 12]. Thus, the biogenic materials were regarded potentially suitable as a catalyst support or as active components in catalysts after modification.

Chemical methods for production of iron oxides use toxic substances, consume much energy, and produce dangerous waste solutions [2, 7, 13–15]. Iron oxide/hydroxide compounds are obtained in nature because of iron biomineralization. Bio-inspired technologies imitate these processes including mediators like microorganisms and phytochemicals. Such methods are realized under ambient conditions and have an ecology friendly character [2, 5, 6, 11, 15–22]. Such a production does not require big investments and additional energy supply because iron bacteria are normal cell metabolites.

The present investigation is concerned with characterization of a hybrid material prepared by deposition of biogenic iron on a fibrous Pd catalyst by cultivation of iron bacteria in selected medium. The following methods were used in the study: X-ray photoelectron spectroscopy (XPS), scanning electron microscopy with electron dispersive analysis (SEM-EDX), infrared spectroscopy (IRS), and low temperature adsorption of nitrogen (Brunauer-Emmett-Teller method). *In situ* diffuse-reflectance infrared spectroscopy (DRIRS) was applied to monitor catalytic tests of the composite in the reaction of CO oxidation.

EXPERIMENTAL

A 0.3%Pd/Al-Si-O fibrous catalyst, synthesized by a sol-gel method [23], reduced at 400°C for 2 h, and then kept in air was used. A Fe-Pd/Al-Si-O sample was obtained by deposition of biogenic iron on a previously prepared 0.3%Pd/Al-Si-O by culti-

---

* To whom all correspondence should be sent.
E-mail: shopska@ic.bas.bg

vation in laboratory conditions of the *Leptothrix* genus of bacteria in a silicon-iron-glucose-peptone elective medium (SIGP [24]) under static conditions at 20°C. The fibrous material was sterilized by autoclaving for 15 min at 1 atm and 120°C and then added to the medium before its infection with the bacteria. After a 36-day period of cultivation, the product was collected through decantation, washed with distilled water, and dried at 40°C for 24 h.

Specific surface area of the studied basic materials was measured according to the BET method (adsorption of N_2 at -196°C) by a NOVA-1200e high-speed gas sorption analyser (Quantachrome Instruments, USA). Prior to measurement, the samples were evacuated at 100°C for 12 h. Pore size distribution was estimated using the BJH method.

XPS measurements were carried out in the UHV chamber of an ESCALAB-MkII (VG Scientific) electron spectrometer with a base pressure of about 1×10^{-10} mbar (1×10^{-9} mbar during measurements) at room temperature. The photoelectron spectra were obtained using unmonochromatized AlKα (hν = 1486.6 eV) radiation. Passing through a 6-mm slit (entrance/exit) of a hemispherical analyser enabled detection of electrons of 20-eV energy by a channeltron. Because of a small signal for Pd 3d, 50-eV pass energy was used. The instrumental resolution, measured as the full width at half maximum (FWHM) of the Ag $3d_{5/2}$ photoelectron peak, was about 1 eV. The energy scale was corrected to the C 1s peak maximum at 285.0 eV for electrostatic sample charging. Processing of the measured spectra included a subtraction of X-ray satellites and Shirley-type background [25]. Peak positions and areas were evaluated by a symmetrical Gaussian-Lorentzian curve fitting. The relative concentrations of the different chemical species were determined based on normalization of the peak areas to their photoionization cross-sections calculated by Scofield [26].

Scanning electron micrographs were recorded on a MIRA3 FE-SEM microscope (TESCAN, Czech Republic) working at accelerating voltage up to 30 kV and magnification up to 1000000x. Energy dispersive X-ray analysis (EDX) (Oxford Instruments, UK) of selected parts of the samples was applied to determine chemical composition and analyse Pd and Fe distribution in the catalysts.

Infrared spectra (IR) of the samples were recorded on a Nicolet 6700 FTIR spectrometer (Thermo Electron Corporation, USA) using the method of dilution of studied material in KBr pellets (0.5% of studied substance). The spectra were collected in the middle region using 50 scans. Catalytic activity in the CO oxidation reaction was studied using a high-temperature vacuum chamber diffuse-reflectance accessory of the spectrometer (Collector II, Thermo Spectra-Tech). Prior to catalytic measurements, the samples were treated in oxidative flow of 25% O_2 in N_2 in T_{room}–200°C's interval in order to clean their surfaces. *In situ* measurements were performed using a reaction flow mixture of 9 vol.% CO, 11.4 vol.% O_2 in nitrogen at 88 $ml.min^{-1}$. The used interval of reaction temperatures was T_{room}–250°C as well as T_{room}–300°C only for 0.3%Pd/Al-Si-O. The temperature was raised consecutively to the necessary reaction temperature at a rate of 10 $deg.min^{-1}$ and held for 30 min at each attained level. This procedure was aimed at achieving constant parameters of the studied system (constant infrared spectra in selected conditions).

RESULTS AND DISCUSSION

BET measurements of the initial 0.3%Pd/Al-Si-O catalyst determined a specific surface area of 29 $m^2.g^{-1}$. Registered pore size distribution revealed a mesoporous material (2–50 nm) displaying a predominant pore size of about 6.5 nm (Fig. 1). Deposition of biogenic iron-containing material slightly changes composite surface properties: the specific surface area of the Fe-Pd/Al-Si-O sample is 37 $m^2.g^{-1}$, while predominant pore sizes are 3.8 and 5.3 nm.

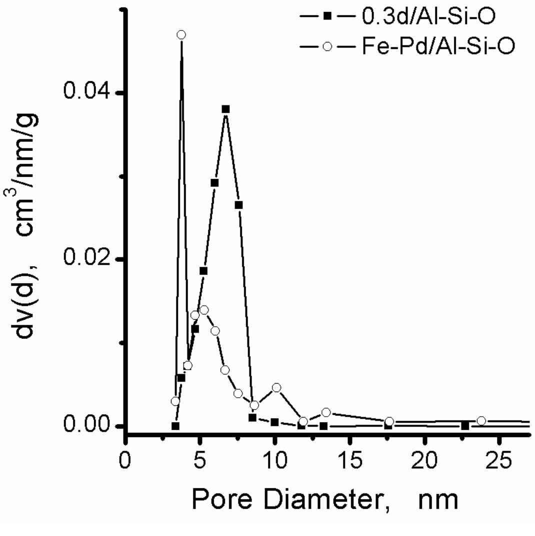


Fig. 1. Pore size distribution in the studied materials.

IR spectra of the samples were characterized by a spectrum drop in the region under 1200 cm^{-1} (Figs. 2 and 3). This feature is attributable to fibre sample morphology and different refractive indices of fibrous material and used matrix. Sets of bands characteristic of alumina and silica supports were registered at 570, 665 cm^{-1} (Al-O), and 570, 798 (Si-O) for the 0.3%Pd/Al-Si-O system and at 550, 583, and 664 cm^{-1} (Al-O), and 380, 583, 790, and 1080 cm^{-1} (Si-O) for the Fe-Pd/Al-Si-O system [27]. The IR spectrum of biogenic material cultivated in the absence of fibrous material proves the formation of γ-FeOOH in SIGP medium. This is shown as inset in figure 3. Characteristic bands of lepidocrocite were registered at 480, 575, 950, 1040, and 1150 cm^{-1} [28–31].

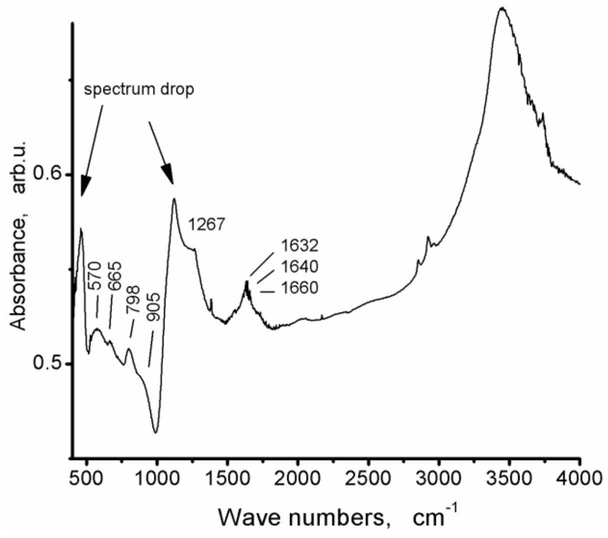


Fig. 2. IR spectrum of 0.3%Pd/Al-Si-O fibrous catalyst.

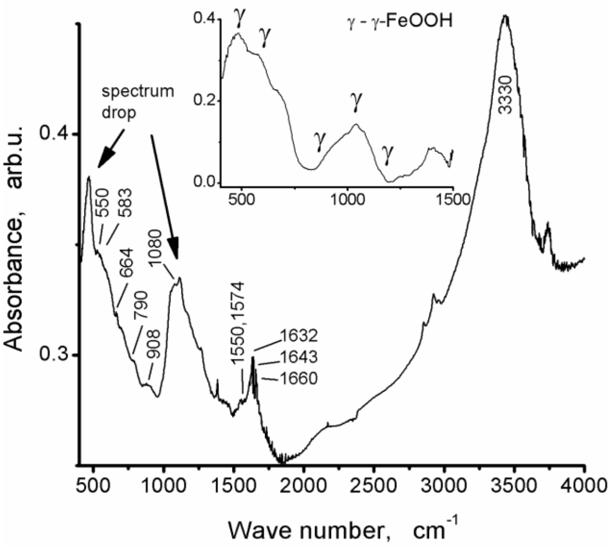


Fig. 3. IR spectrum of Fe-Pd/Al-Si-O sample. Inset – IR spectrum of biogenic iron-containing material obtained in SIGP medium in absence of 0.3%Pd/Al-Si-O fibrous catalyst.

XPS measurements were performed to monitor elements present on the surface and their oxidation states. XP spectra of the substrate revealed occurrence of Al–O (probably Al_2O_3), Si=O (characteristic of SiO_2), and Si–O species. Our XPS analysis was focused on the Pd and Fe catalytic materials. It was observed that palladium was present in two different oxidation states on the surface of an as-received Pd/Al-Si-O sample (prereduced and kept in air), namely, Pd^{2+} and Pd^0 at a Pd^{2+}/Pd^0 ratio of 0.4 (Fig. 4). Comparison with data acquired after calcination at 300°C for 30 min disclosed Pd redistribution giving rise to about a 20%-increase in surface concentration of the palladium (from 2.5×10^{-3} to 3×10^{-3} at.%). Both forms of Pd were present but the Pd^{2+}/Pd^0 ratio was changed to two.

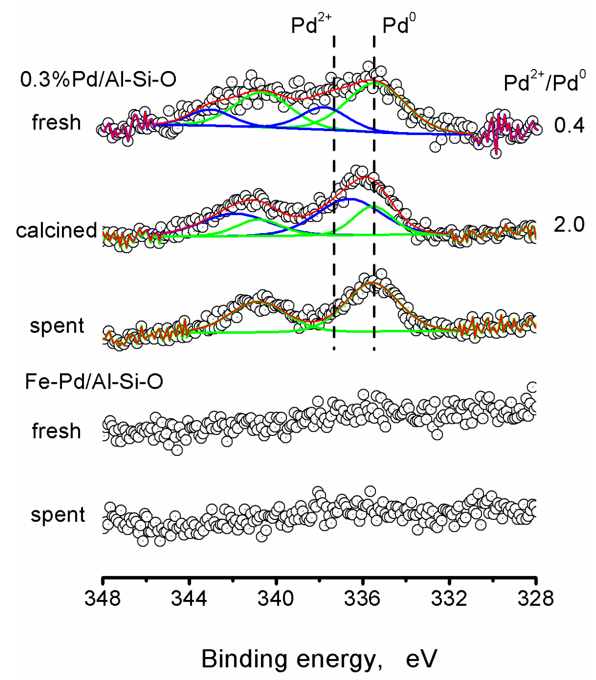


Fig. 4. High resolution XP spectra of Pd and respective Pd^{2+}/Pd^0 ratios.

High resolution XP spectra of as received fresh and used Fe-Pd/Al-Si-O samples demonstrated the presence of iron in two forms, $Fe^{3+} \approx Fe^{2+}$ at a Fe/(Al+Si) ratio of about 10.5×10^{-2} (Fig. 5).

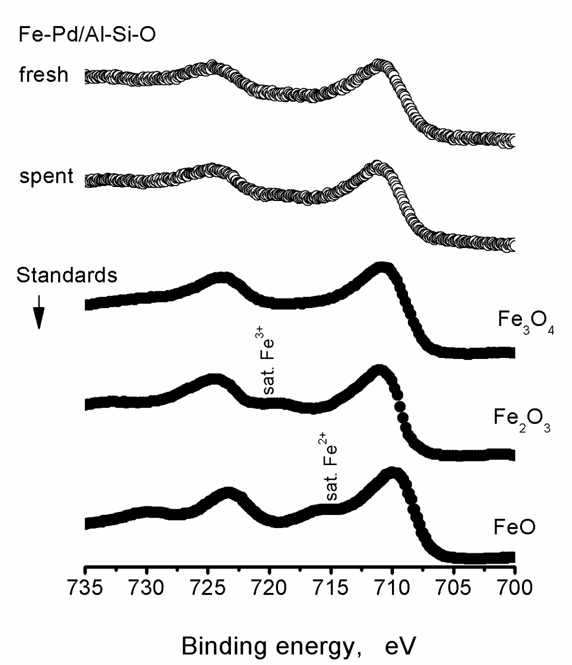


Fig. 5. High resolution XP spectra of Fe in Fe-Pd/Al-Si-O sample and standards used.

Iron occurrence in both forms was concluded after comparison of the measured XP spectra with standard spectra of Fe^{2+} and Fe^{3+} and a mixture of equal amounts of Fe^{2+} and Fe^{3+} [32]. A very low amount of palladium, within the limits of instrument error, was observed (two bottom lines in figure 4). Additionally, we observed contaminations of C, Cl, and F from preparation procedures, which are not discussed.

The studied samples were tested in the reaction of CO oxidation. The process was monitored *in situ* by diffuse-reflectance infrared spectroscopy. Collected spectra are shown in figures 6 and 7. CO appears in all spectra as a doublet centred at 2144/2142 cm^{-1} [27]. The two wings are not symmetrical, which directs to availability of adsorbed form of CO on the surface of both samples. CO_2 is a reaction product and it was registered by a band at about 2333/2335 cm^{-1} [27]. On increasing the reaction temperature, the CO characteristic doublet was decreased in intensity whereas the CO_2 band intensity was increased. The observed changes were more pronounced in the case of the 0.3%Pd/Al-Si-O system. A general observation in both studied systems was a gradual loss of OH surface coverage, which was well visible above 150°C through an intensity decrease of the band at about 3400 cm^{-1} [27].

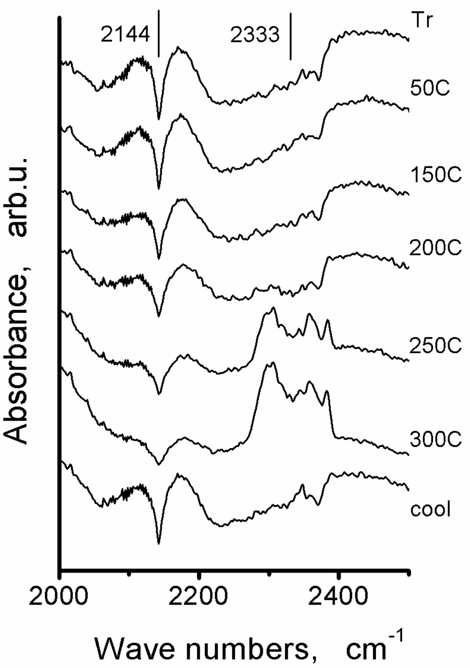


Fig. 6. In situ DRIR spectra collected during CO oxidation in the presence of 0.3% Pd/Al-Si-O fibrous catalyst.

Catalytic activity of the used materials was evaluated through changes in CO gas-phase band integral intensity on increasing the temperature. A 2.5-fold decrease of the carbon monoxide in the reaction mixture was measured over the Pd/Al-Si-O catalyst at 250°C. Fe-Pd/Al-Si-O manifested nearly a 2-fold decrease at this temperature. An additional 2-fold decrease was determined for 0.3%Pd/Al-Si-O after a temperature rise from 250 to 300°C.

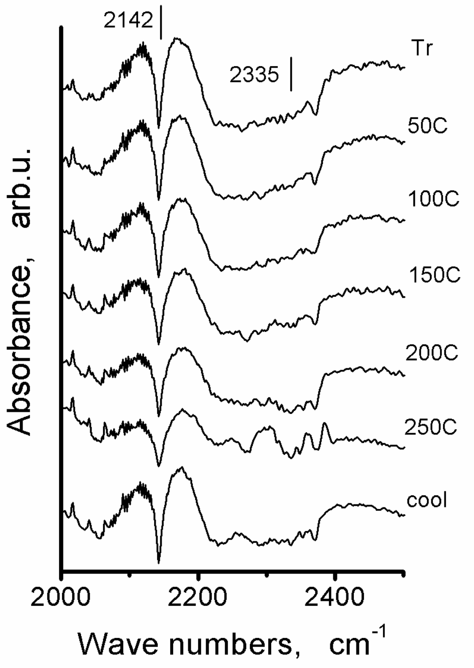


Fig. 7. In situ DRIR spectra collected during CO oxidation in presence of biogenic iron-containing Fe-Pd/Al-Si-O sample.

Figure 8 shows calculated CO conversions in conducted experiments. 0.3%Pd/Al-Si-O demonstrates an almost constant increase in activity with reaction temperature rise. Biogenic iron-containing material demonstrates a slightly lower activity despite that it contains two potentially active components in oxidation reaction, namely Pd and the redox couple of Fe^{3+}-Fe^{2+}. Actually, the Fe-Pd/Al-Si-O system manifests a CO conversion at 50°C showing that the biogenic iron-containing material has intrinsic activity. The 0.3%Pd/Al-Si-O sample has very low activity at 50°C and becomes more active at higher temperatures. The obtained results correspond to a necessity of Pd to be converted in active form. Thus, the experiment indicates that the initial material changes with time on stream. Formation of more or a less active iron-containing phase in the Fe-Pd/Al-Si-O sample can be supposed to occur in the interval 150–200°C giving rise to a shift of the curve but keeping the slope at higher temperatures. Our previous investigations on the catalytic activity of biogenic iron-containing materials showed that FeOOH modifications were converted to respective Fe_2O_3 under these conditions [12].

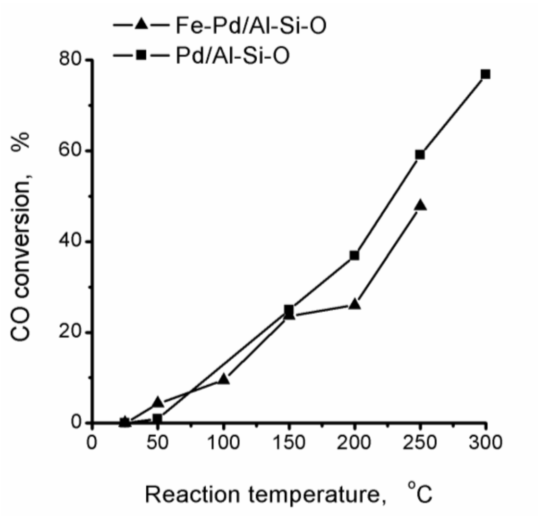
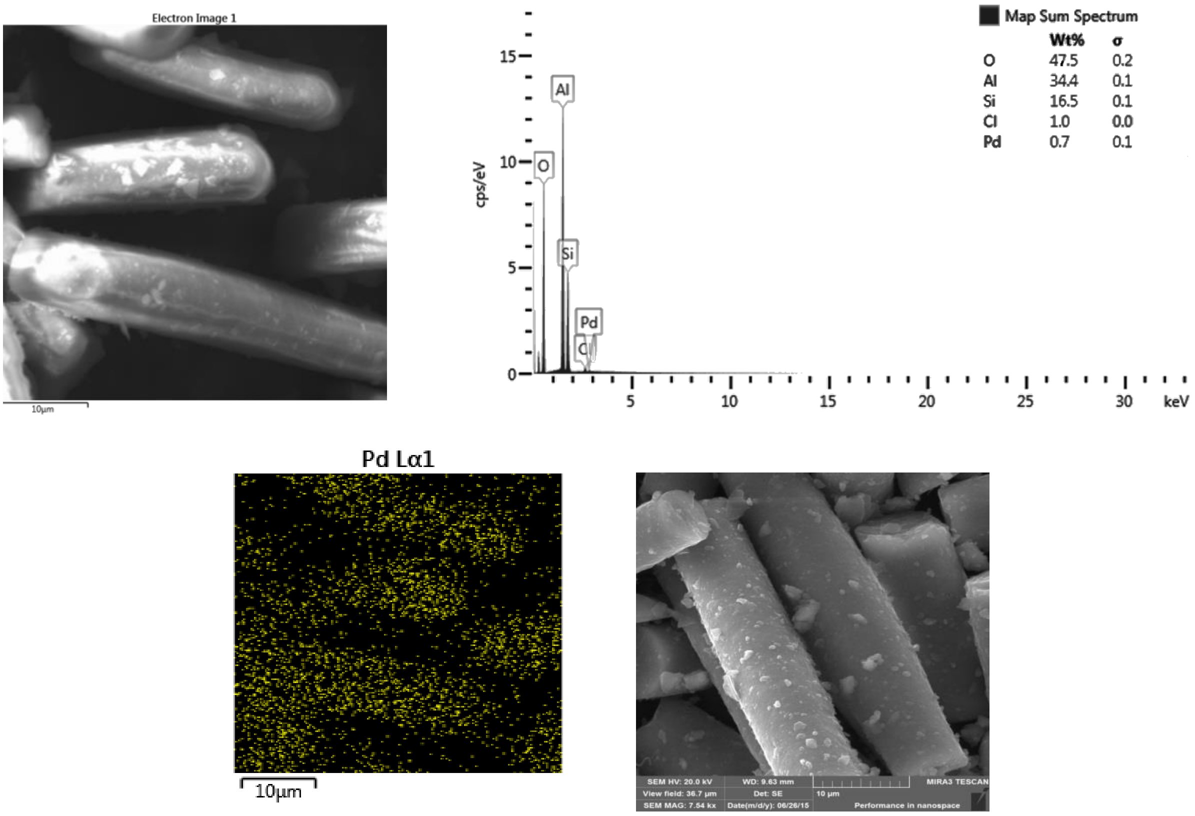


Fig. 8. CO conversion on studied samples.

Spent samples (after catalytic measurements) were studied by SEM and XPS. SEM images (Figures 9 and 10) show broken fibres (visible as dark area) with deposited phases of Pd/PdO and biogenic iron (visible as light area). EDX study showed uniform distribution of Pd on the surface in both tested catalyst samples. However, iron was unevenly distributed, sometimes as being predominantly concentrated on fibre edges. The latter observation corresponds to very small changes of specific surface area after deposition of biogenic iron as determined by BET measurements of the hybrid material. As it is shown on both sample map sum spectra, Pd concentration is 0.7(1) wt.% and that of Fe is 18.8%. These results show that the expected composition of the hybrid sample has been achieved. SEM-EDX analyses revealed a Pd concentration value, which is not in contradiction of XPS data. A registered very low amount of Pd, within the error limits of the ESCALAB instrument, is due to method constraints. Both methods derive information on sample surface from different depth. Sampling depth of XPS analysis is up to 10 nm (depending on the kinetic energy of detected electron), however, for SEM-EDX it is up to 5 µm. Thus, XPS results show that Pd is 'buried' in the alumina-silica matrices of the fibrous material [23] and additionally by deposited biogenic iron as the depth is more than 10 nm. Therefore, Pd is detectable by the SEM-EDX method but not completely by the XPS.

In this context, a lower catalytic activity of the hybrid material can be explained by partial pore and Pd surface blocking, as seen by iron surface concentration in the composite material, which is more than one order of magnitude higher than that of palladium.



Map Sum Spectrum

	Wt%	σ
O	47.5	0.2
Al	34.4	0.1
Si	16.5	0.1
Cl	1.0	0.0
Pd	0.7	0.1

Fig. 9. Electron images of spent 0.3% Pd/Al-Si-O fibrous material, EDX map sum spectrum and EDX map of Pd(Lα1) distribution.

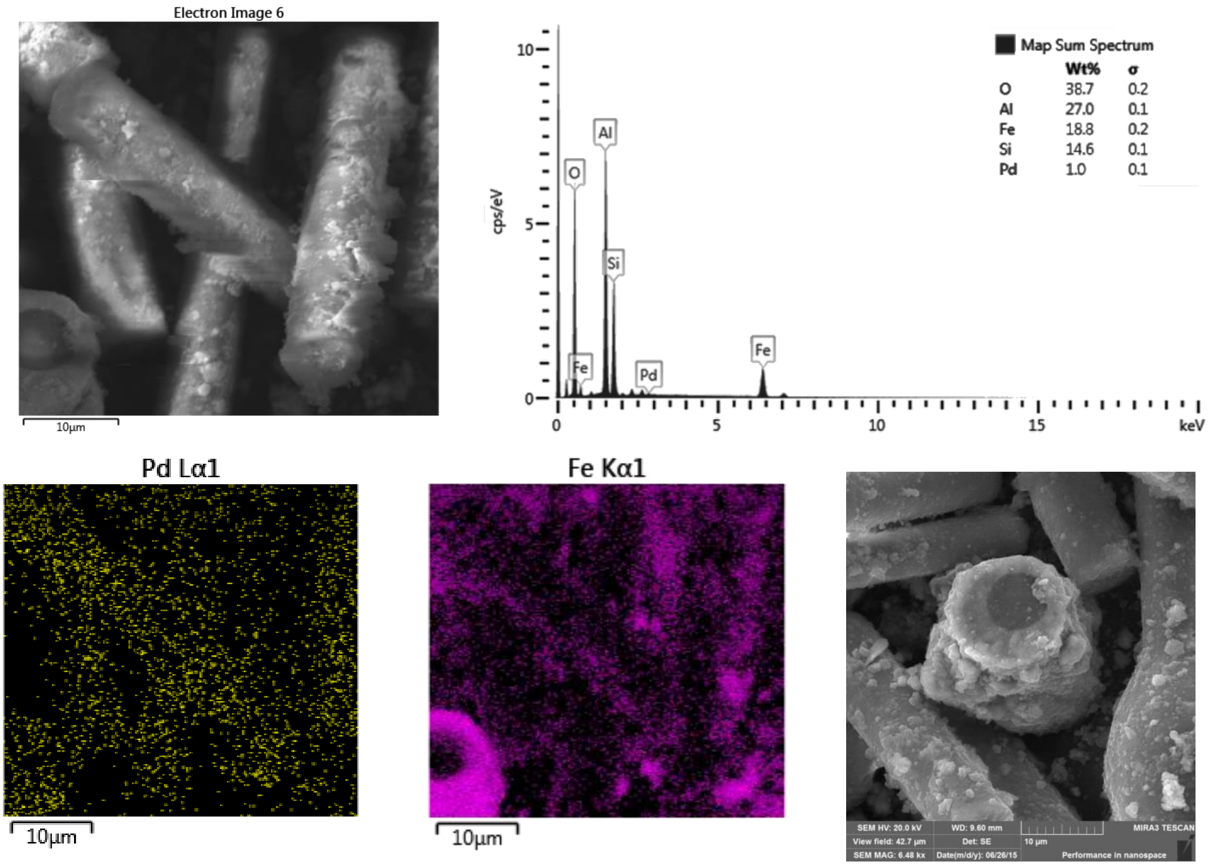


Fig. 10. Electron images of spent biogenic iron-containing Fe-Pd/Al-Si-O sample, EDX map sum spectrum and EDX maps of Pd(Lα1) and Fe(Kα1) distribution.

High resolution XPS analysis of the tested samples revealed existence of Pd^0 only on the surface of the 0.3%Pd/Al-Si-O sample at a Pd/(Al+Si) ratio of 2.3×10^{-3}, which was similar to that in the fresh sample (Fig. 4). Comparative XPS investigation of the fresh and spent 0.3%Pd/Al-Si-O system proved transition of Pd^{2+} to Pd^0. This supports a process of Pd transformation into an active form with time on stream at elevated temperatures due to reduction of Pd by CO in the mixture. The fibrous Fe-Pd/Al-Si-O catalyst with deposited biogenic iron was almost unchanged after the catalytic test. Palladium amount was preserved (within the error limits of the XP spectrometer) at a Pd/(Al+Si) ratio of 1.7×10^{-3}. Iron was present in two forms on the surface, $Fe^{3+} \approx Fe^{2+}$, at a Fe/(Al+Si) ratio of 11.4×10^{-2} (Fig. 5).

The results obtained on studying spent samples by SEM and XPS allow concluding that a close catalytic behaviour of both the studied systems is due to stable dispersion of Pd and iron on the catalyst surface that is preserved during/after reaction.

CONCLUSIONS

This study indicated that the catalytic activity of both 0.3%Pd/Al-Si-O and Fe-Pd/Al-Si-O systems was essentially due to palladium. Despite the presence of a Fe^{3+}-Fe^{2+} couple, which is active in oxidation reactions, no synergism was observed. Similarities between the catalytic performances of the studied systems could be explained by uniform and stable distribution of palladium and iron on the surface, which is preserved under reaction conditions.

Acknowledgements: The authors are grateful to the Bulgarian Science Fund for financial support by project T02-17/2014 as well as to the Bulgarian Academy of Sciences and the Slovak Academy of Sciences for support through the bilateral project 'Mechanochemical synthesis – an ecological friendly process in the production of materials for photo-catalytic air and water purification'. The supply of Pd catalyst by Dr. L. Spasov and Dr. Ch. Bonev (Institute of Catalysis), and the deposition of biogenic material by Prof. V. Groudeva and Dr. M. Iliev (Sofia University) is greatly acknowledged. M.F. and J.B. also thank VEGA (2/0097/13) and APVV (14-0103) for support of their work.

REFERENCES

1. H. Jung, H. Park, J. Kim, J.-H. Lee, H.-G. Hur, N. V. Myung, H. Choi, *Environ. Sci. Technol.*, **41**, 4741 (2007).

2. B. Kazprzyk-Hordern, M. Ziolek, J. Nawrocik, *Appl. Catal. B: Environ.*, **46**, 639 (2003).
3. K. Mandai, T. Korenaga, T. Ema, T. Sakai, M. Furutani, H. Hashimoto, J. Takada, *Tetrahedron Lett.*, **53**, 329 (2012).
4. T. Ema, Y. Miyazaki, I. Kozuki, T. Sakai, H. Hashimoto, J. Takada, *Green Chem.*, **13**, 3187 (2011).
5. G. E. Hoag, J. B. Collins, J. L. Holcomb, J. R. Hoag, M. N. Nadagouda, R. S. Varma, *J. Mater. Chem.*, **19**, 8671 (2009).
6. T. Shahwan, S. Abu Sirriah, M. Nairat, E. Boyaci, A. E. Eroglu, T. B. Scott, K. R. Hallam, *Chem. Eng. J.*, **172**, 258 (2011).
7. R. A. Maithreepala, R.-A. Doong, *Chemosphere*, **70**, 1405 (2008).
8. B. Kumar, K. Smita, L. Cumbal, A. Debut, *J. Saudi Chem. Soc.*, (2014), http://dx.doi.org/10.1016/j.jscs.2014.01.003
9. A. Alharthi, R. A. Blackley, T. H. Flowers, J. S. J. Hergreaves, I. D. Pulford, J. Wigzell, W. Zhou, *J. Chem. Technol. Biotechnol.*, (2014), doi:10.1002/jctb.4434
10. T. Sakai, Y. Miyazaki, A. Murakami, N. Sakamoto, T. Ema, H. Hashimoto, M. Furutani, M. Nakanishi, T. Fujiia, J. Takada, *Org. Biomol. Chem.*, **8**, 336 (2010).
11. M. Shopska, Z. Cherkezova-Zheleva, D. Paneva, M. Iliev, G. Kadinov, I. Mitov, V. Groudeva, *Cent. Eur. J. Chem.*, **11**, 215 (2013).
12. M. Shopska, D. Paneva, G. Kadinov, S. Todorova, M. Fabian, I. Yordanova, Z. Cherkezova-Zheleva, I. Mitov, *Reac. Kinet. Mechan. Catal.* (submitted).
13. K. B. Narayanan, N. Sakthivel, *Adv. Colloid Interface Sci.*, **156**, 1 (2010).
14. H. Jung, J.-W. Kim, H. Choi, J.-H. Lee, H.-G. Hur, *Appl. Catal. B: Environ.*, **83**, 208 (2008).
15. A. B. Seabra, P. Haddad, N. Duran, *IET Nanobiotechnol.*, **7**, 90 (2013).
16. H. Hashimoto, S. Yokoyama, H. Asaoka, Y. Kusano, Y. Ikeda, M. Seno, J. Takada, T. Fujii, M. Nakanishi, R. Murakami., *J. Magnet. Magnet. Mater.*, **310**, 2405 (2007).
17. B. Xin, D. Zhang, X. Zhang, Y. Xia, F. Wu, S. Chen, L. Li, *Bioresour. Technol.*, **100**, 6163 (2009).
18. J. A. Rentz, I. P. Turner, J. L. Ullman, *Water Res.*, **43**, 2029 (2009).
19. R. Cornell, U. Schwertmann, Iron Oxides, Wiley-VCH Verlag, Weinheim, Germany, 2003.
20. R. W. Fitzpatrick, R. Naidu, P. G. Self, in: Biomineralization Processes of Iron and Manganese - Modern and Ancient Environments, Catena Suppl. 21, H. C. W. Skinner, R. W. Fitzpatrick Eds., Catena Verlag, Reiskirchen, Germany, 1992, p. 263.
21. D. A. Ankrah, E. G. Sogaard, 13th Int. Water Technol. Conf., IWTC 13, 2009, Hurghada, Egypt, p. 999.
22. A. Alharthi, R. A. Blackley, T. H. Flowers, J. S. J. Hergreaves, I. D. Pulford, J. Wigzell, W. Zhou, *J. Chem. Technol. Biotechnol.*, **89**, 1317 (2014).
23. L. Spasov, P. Dimitrov, Ch. Vladov, V. Zhelyazkov, Ch. Bonev, L. Petrov, in: Heterogeneous Catalysis (Proc. 8th Int. Symp. Heterogen. Catal., Varna, 5–9 October 1996), Institute of Catalysis, Bulgarian Academy of Sciences, Sofia, 1996, p. 751.
24. M. Sawayama, T. Suzuki, H. Hashimoto, T. Kasai, M. Furutani, N. Miyata, H. Kunoh, J. Takada, *Curr. Microbiol.*, **63**, 173 (2011).
25. D. Shirley, *Phys. Rev. B*, **5**, 4709 (1972).
26. J. H. Scofield, *J. Electron Spectrosc. Relat. Phenom.*, **8**, 129 (1976).
27. L. H. Little, Infrared Spectra of Adsorbed Species, Academic Press Inc., London, New York, 1966.
28. A. U. Gehring, A. M. Hofmeister, *Clays Clay Miner.*, **42**, 409 (1994).
29. N. Lazaroff, W. Sigal, A. Wasserman, *Appl. Environ. Microbiol.*, **43**, 924 (1982).
30. S. Music, A. Saric, S. Popovic, K. Nomura, T. Sawada, *Croat. Chem. Acta*, **73**, 541 (2000).
31. B. Weckler, H. D. Lutz, *Eur. J. Solid State Inorg. Chem.*, **35**, 531 (1998).
32. S. Gota, E. Guiot, M. Henriot, M. Gautier-Soyer, *Phys. Rev. B*, **60**, 14387 (1999).

СИНТЕЗ НА КАТАЛИЗАТОР Fe-Pd/Al-Si-O СЪДЪРЖАЩ БИОГЕННО ЖЕЛЯЗО ЧРЕЗ КУЛТИВИРАНЕ НА БАКТЕРИИ ОТ РОДА *LEPTOTHRIX*

М. Г. Шопска[1]*, Г. Б. Кадинов[1], Я. Бриянчин[2], Ил. Д. Йорданова[1], Хр. Г. Колев[1], М. Фабиан[2]

[1] Институт по катализ, Българска академия на науките, ул. „Акад. Г. Бончев", Бл. 11, 1113 София, България
[2] Институт по геотехника, Словашка академия на науките, ул. „Ватсонова" № 45, 040 01 Кошице, Словакия

Постъпила на 15 септември 2015 г.; Преработена на 4 ноември 2015 г.

(Резюме)

Чрез култивиране на бактерии от рода *Leptothrix* в среда силиций-желязо-глюкоза-пептон е отложено биогенно желязо върху влакнест катализатор 0.3%Pd/Al-Si-O. Така е получен композитен материал Fe-Pd/Al-Si-O. Методите инфрачервена спектроскопия, сканираща електронна микроскопия (с енергийно дисперсивен рентгенов анализ), нискотемпературна адсорбция на азот и рентгенова фотоелектронна спектроскопия бяха използвани при охарактеризиране на изследваните образци. Установено бе, че хибридният материал съдържа биогенен лепидокрокит, γ-FeOOH. Каталитичната активност на образците бе изследвана в реакцията на окисление на CO. Поведението на системите бе регистрирано *in situ* чрез дифузно-отражателна инфрачервена спектроскопия. Каталитичните изпитания целяха оценка на качествата на системи, получени при модифициране на биогенно желязо. Влакнестият катализатор 0.3%Pd/Al-Si-O и хибридният материал Fe-Pd/Al-Si-O показаха подобни каталитични отнасяния. Това се дължи на еднородното и стабилно разпределение на паладия и желязото по повърхността на образците. Въпреки, че на синтезирания композит е присъща каталитичната активност при ниски температури, регистрираната активност в изследвания интервал от реакционни температури до голяма степен се дължи на паладия.