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Book of papers

10th Slovenian-Serbian-Croatian Symposium on Zeolites

National Institute of Chemistry, Ljubljana, Slovenia, May 23 – 25, 2024

Editors: Alenka Ristić, Marjana Gantar Albreht, Ana Palčič and Sanja Jevtić

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PREFACE

On behalf of the local and organizing committee, I would like to welcome you to the 10th Slovenian-Serbian-Croatian Symposium on Zeolites, which will take place from 23 to 25 May 2024 at the National Institute of Chemistry of Slovenia in Ljubljana, Slovenia.

At the conference, an impressive number of distinguished researchers, young researchers and students will present the latest scientific achievements on natural and synthetic zeolites as well as related nanoporous materials such as aluminophosphates, metalo-organic and zeolitic imidazolate frameworks as well as organic-inorganic hybrid materials for environmental, energy, biomedical and microbiological applications. In addition to two plenary lectures, 3 keynote lectures and 3 invited lectures participants will have the opportunity to present their work in oral and short-oral presentations.

We are very pleased to welcome the recognized plenary speakers Prof. Nevenka Rajić (Nikola Tesla University, Belgrade, Serbia) and Assoc. Prof. Monique van der Veen from Delft University of Technology (The Netherlands), the keynote speakers Srna Stojanović (Faculty of Physical Chemistry, Belgrade, Serbia), Assoc. Prof. Tomislav Ivanković (Faculty of Science, Zagreb, Croatia) and Assoc. Prof. Sebastijan Kovačič (National Institute of Chemistry, Ljubljana, Slovenia). The invited lecturers are Assoc. Prof. Miha Grilc (National Institute of Chemistry, Ljubljana, Slovenia), Assoc. Prof. Ivana Grčić (Faculty of Geotechnical Engineering, Varaždin, Croatia) and Professor Emeritus Jean-Pierre Gilson (Laboratory for Catalysis & Spectrochemistry ENSICAEN, Caen, France).

An exhibition of laboratory and process equipment will also be organized during the conference. I am pleased that we have been able to attract 8 suppliers of research equipment to exhibit at this conference. I would like to take this opportunity to thank the companies, Altium, AstrinexLab, Chemass, Donau Lab, Kobis, Parr Instrument, Sanolabor, Silkem, Analysis Adria, Primatron, Bia and Surface Measurements Systems for sponsoring this event. Two sponsor presentations will be given by AstrinexLab and Altium.

Finally, I would like to thank the members of the organizing committee for their cooperation in defining the conference profile and selecting the plenary and keynote speakers and to you for your contribution to this successful conference.

Yours faithfully,

Alenka Ristić

Chair of the Organizing Committee

PLENARY LECTURES

PL1: [On the Applications of the Natural Clinoptilolite from Serbian deposits](#)

[Nevenka Rajić](#)

The University "UNION – Nikola Tesla", Belgrade, Serbia

PL2: [Metal-organic frameworks: a dynamic playground for water and energy harvesting](#)

[Monique A. van der Veen](#)

Department of Chemical Engineering, Delft University of Technology, the Netherlands

KEYNOTE LECTURES

KL1: Porous Organic-Inorganic Hybrid Materials: New synthetic Approaches, Engineering and Applications

Sebastijan Kovačič^{1, 2}, Matjaž Mazaj¹, Zoran Novak²

¹ National Institute of Chemistry, Ljubljana, Slovenia

² Faculty of Chemistry and Chemical Engineering, University of Maribor, Slovenia

KL2: Microbiological applications of natural zeolite

Tomislav Ivanković¹, Jasna Hrenović¹, Nevenka Rajić², Jelena Dikić³, Sabine Rolland du Roscoat⁴

¹ Faculty of Science, University of Zagreb, Croatia

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³ Innovation centre of the Faculty of Technology and Metallurgy, University of Belgrade, Serbia

⁴ Laboratoire 3SR, Université Grenoble Alpes, Domaine Universitaire, France

KL3: The efficiency of TiO₂/zeolites for photocatalytic degradation of different pollutants from aqueous solution

Srna Stojanović¹, Milan Damjanović², Marija Lješević³, Vladimir Bešković⁴, Vladislav Rac⁵, Ljiljana Damjanović-Vasilić¹

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⁵ University of Belgrade – Faculty of Agriculture, Belgrade, Serbia

INVITED LECTURES

IL1: Modeling of sorption and surface reaction microkinetics for vapor-phase hydro(deoxy)genation of levulinic acid over Ni/ZSM-5

Miha Grilc^{1,2}, Žan Lavrič^{1,2}, Hue-Tong Vu¹, Andrii Kostyniuk¹, Nataša Zabukovec Logar^{1,2}, Petar Djinović^{1,2}, Nataša Novak Tušar^{1,2}, Blaž Likozar¹

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² University of Nova Gorica, Slovenia

IL2: Industrial Deployment of a Zeolite Catalyst: Past, Present & Future

Jean-Pierre Gilson

Laboratory for Catalysis & Spectrochemistry ENSICAEN, Caen, France

IL3: Zeolites in photocatalytic treatment of water

Ivana Grčić

Faculty of Geotechnical Engineering, University of Zagreb, Varaždin, Croatia

ORAL LECTURES

O1: Comparison of zeolite-based hybrid processes in wastewater treatment – preliminary study

Ladislav Vrsalović, Nediljka Vukojević Medvidović, Sandra Svilović, Senka Gudić, Nikolina Mileusnić
Faculty of Chemistry and Technology, University of Split, Croatia

O2: Preparation of bioparticles for phosphorus removal from wastewater in real scale

Jasna Hrenović¹, Stanislaw Lazarek², Darko Tibljaš¹

¹ *Faculty of Science, University of Zagreb, Croatia*

² *BeAquaAgain AB, Malmo, Sweden*

O3: Zeolitic imidazolate framework materials for potential adsorption thermal battery applications

Jan Marčec^{1,2}, Alenka Ristić¹

¹ *National Institute of Chemistry, Ljubljana, Slovenia*

² *University of Nova Gorica, Slovenia*

O4: The Influence of Working Fluids on the Heat Storage Performance of ZIF-93

Ciara Byrne^{1,2}, Katja Vodlan³, Connor Hewson⁴, Paul Iacomi⁴, Amalija Golobič³, Nataša Zabukovec Logar^{1,2}

¹ *National Institute of Chemistry, Ljubljana, Slovenia*

² *University of Nova Gorica, Slovenia*

³ *University of Ljubljana, Slovenia*

⁴ *Surface Measurement Systems, London, United Kingdom*

O5: Water clusters and linde type-A aluminophosphate: interplay between structural order and disorder

Janez Volavšek¹, Suzana Mal¹, Matjaž Mazaj¹, Alenka Ristić¹, Anže Hubman¹, Franci Merzel¹, Nataša Zabukovec Logar^{1,2}, Andraž Krajnc¹

¹ *National Institute of Chemistry, Ljubljana, Slovenia*

² *University of Nova Gorica, Slovenia*

O6: A comprehensive overview of the global zeolites market

Karmen Margeta, Anamarija Farkaš

Institute for Development and International Relations, Zagreb, Croatia

O7: NICS-24: a new microporous MOF for selective CO₂ capture

Andreas Puškarić^{1,2}, Matjaž Mazaj², Nataša Zabukovec Logar²

¹ *Ruđer Bošković Institute, Zagreb, Croatia*

² *National Institute of Chemistry, Ljubljana, Slovenia*

O8: Routes towards preparation of pure nickel zeolitic imidazolate frameworks

Aljaž Škrjanc^{1,2}, Dominik Jankovič³, Anton Meden³, Matjaž Mazaj¹, Erik Svensson Grape⁴, Martin Gazvoda³, Nataša Zabukovec Logar^{1,2}

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² *University of Nova Gorica, Slovenia*

³ Faculty of Chemistry and Chemical Technology, University of Ljubljana, Slovenia

⁴ Department of Materials and Environmental Chemistry, Stockholm University, Sweden

O9: Transformations and synthesis of zeolites upon exerting mechanical force at elevated temperature

Mladenka Jurin¹, Sanja Bosnar¹, Natalija Pantalon Juraj², Nikola Jakupec¹, Martin Etter³, Krunoslav Užarević², Ana Palčič¹

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³ Deutsches Elektronen-Synchrotron (DESY), Hamburg, Germany

O10: Thermal and visible light-assisted CO₂ hydrogenation to CO over an industrial copper based catalyst

Giovanni Cardolini Rizzo¹, Miha Okorn^{2,3}, Kristijan Lorber², Nataša Novak Tušar^{2,3} and Petar Djinović^{2,3}

¹ Politecnico di Torino, Italy

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³ University of Nova Gorica, Slovenia

O11: Bimetal (Cu/Fe) modified mesoporous silica as an efficient fenton-like catalyst for bisphenol A removal

Andraž Šuligoj^{1,2}, Ksenija Maver¹, Albin Pintar¹, Nataša Novak Tušar¹

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² Faculty of Chemistry and Chemical Technology, University of Ljubljana, Slovenia

O12: Exploring the diverse applications of TiO₂+PT catalysts

Gregor Žerjav, Albin Pintar

National Institute of Chemistry, Ljubljana, Slovenia

O13: MOFs grown within the high-internal emulsion polymer support: new adsorbents for CO₂ capture

Matjaž Mazaj¹, Sebastijan Kovačič^{1,2}, Nataša Zabukovec Logar^{1,3}

¹ National Institute of Chemistry, Ljubljana, Slovenia

² University of Maribor, Slovenia

³ University of Nova Gorica, Slovenia

SHORT ORAL LECTURES

SO1: The preliminary study on the natural zeolite as a potential carrier of tetracycline

Danina Krajišnik¹, Snežana Uskoković-Marković², Aleksandra Daković³, Marija Marković³

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³ Institute for Technology of Nuclear and Other Mineral Raw Materials (ITNMS), Belgrade, Serbia

SO2: Natural zeolite clinoptilolite and its sodium form in treatment of zinc contaminated water – how effective are they?

Ivona Nuić, Sara Jurkić, Ivana Zelić, Marin Ugrina, Marina Trgo
Faculty of Chemistry and Technology, University of Split, Croatia

SO3: Application of modified natural zeolite phillipsite for removal of ochratoxin A

Marija Marković¹, Aleksandra Daković¹, Danina Krajišnik², Milica Ožegović¹, George E. Rottinghaus³, Bruno de Gennaro⁴, Mariano Mercurio⁵

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⁴ *DICMAPI, University of Naples, Italy*

⁵ *Department of Sciences and Technology, University of Sannio, Benevento, Italy*

SO4: Natural materials as carriers of microbial consortium

Blanka Dadić¹, Tomas Corak Grdovic¹, Zorana Kovacevic², Jasna Hrenovic¹, Tomislav Ivankovic¹

¹ *Faculty of Science, Department of Biology, University of Zagreb, Croatia*

² *Faculty of Textile Technology, University of Zagreb, Croatia*

SO5: Phosphorus retention in peat amended with iron-containing clinoptilolite

Jelena Pavlović¹, Tore Krogstad², Nevenka Rajić³

¹ *Institute of Soil Science, Belgrade, Serbia*

² *Norwegian University of Life Sciences, Aas, Norway*

³ *The University "UNION – Nikola Tesla", Belgrade, Serbia*

SO6: Synthesis of zeolite and carbon-zeolite composite from waste silica fumes

Saša Zeljković¹, Toni Ivas²

¹ *Faculty of Natural Sciences and Mathematics, University of Banja Luka, Bosnia and Herzegovina*

² *Swiss Federal Laboratories for Materials Science and Technology – EMPA, Thun, Switzerland*

SO7: Mineralogical, micro-structural and mechanical properties of lime mortars containing natural zeolite

Marina Škondrić

Faculty of Civil Engineering, University of Belgrade, Serbia

SO8: Novel zinc oxalate-triazolate framework for selective low-concentration CO₂ capture

Klara Klemenčič¹, Andreas Puškarić^{1,2}, Matjaž Mazaj¹

¹ *National Institute of Chemistry, Ljubljana, Slovenia*

² *Ruđer Bošković Institute, Zagreb, Croatia*

SO9: Copper's story in ZIF-62: surface or structure?

Marija Švegovec^{1,2}, Andraž Krajnc¹

¹ *National Institute of Chemistry, Ljubljana, Slovenia*

² *University of Nova Gorica, Slovenia*

SO10: In situ PXRD measurements of mechanochemically induced interzeolite conversion – advantages and drawbacks

Nikola Jakupec¹, Erik Uran², Martin Etter³, Krunoslav Užarević¹ Ana Palčić¹

¹ Ruđer Bošković Institute, Zagreb, Croatia

² Jožef Stefan Institute, Ljubljana, Slovenia

³ Deutsches Elektronen-Synchrotron, Hamburg, Germany

SO11: Dehydration performance of a novel solid solution library of mixed and supported tutton salts as thermochemical heat storage materials

Jakob Smith, Anton Larchier, Weinberger Peter, Werner Andreas

TU Wien, Technische Universität Wien Lehartrakt, Österreich

SO12: Exploring the impact of plasmonic metals on the properties of plasmonic metal + TiO₂ catalysts

Špela Slapničar, Gregor Žerjav, Albin Pintar

National Institute of Chemistry, Ljubljana, Slovenia

SO13: Comparison of potential of fruit processing by-products and natural zeolite clinoptilolite in treatment of ZINC(II)-contaminated water

Sunčica Mileta¹, Ivona Nuić², Ana Palčić³, Marin Ugrina², Ivana Raguž²

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² Faculty of Chemistry and Technology, University of Split, Croatia

³ Ruđer Bošković Institute, Zagreb, Croatia

SO 14: The sorption and retention capability of natural and iron-modified zeolites toward manganese ions

Marin Ugrina, Lorena Tomić, Ana Terzić, Ivona Nuić, Marina Trgo

Department of Environmental Engineering, Faculty of Chemistry and Technology, University of Split, Croatia

SO15: Synthesis and characterization of hydroxyapatite/clinoptilolite adsorbent for removal of organic pollutants

Katarina Sokić¹, Teodora Taškov¹, Jelena Dikić², Ljiljana Tolić Stojadinović², Sanja Jevtić¹

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SO16: Solid-state nuclear magnetic resonance used for operando studies of Na-ion batteries

Matej Gabrijelčič^{1,2}, Blaž Tratnik¹, Gregor Kapun¹, Elena Tchernychova¹, Nataša Zabukovec Logar¹, Andraž Krajnc¹, Robert Dominko^{1,3,4}, Alen Vižintin¹

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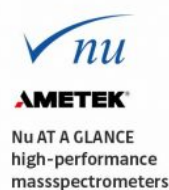
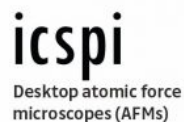
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PHOSPHORUS RETENTION IN PEAT AMENDED WITH IRON-CONTAINING CLINOPTILOLITE

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ABSTRACT

Although phosphorus (P) is a crucial plant nutrient, it is associated with environmental problems such as eutrophication. The effect of Fe(III)-containing natural clinoptilolite (FeCLI) on P retention in peat soil, an acidic soil typically high in organic matter, was studied by a batch method. The effect was investigated both in pure peat and lime-amended peat. The peat amount and the rate of lime influenced the retention efficiency of FeCLI. The acidity of both pure peat and peat that have been amended with lime was decreased in the presence of FeCLI. All obtained results suggested that FeCLI can be considered as a beneficial soil supplement to support both soil fertility and water preservation.

Key words: natural zeolite, iron–clinoptilolite, peat, organic soil, phosphorus.

INTRODUCTION

Because it affects soil fertility and environmental pollution, mitigation of P loss from soils has attracted much attention. One obvious reason for P loss is the intense threat to peat soils, an acidic soil typically high in organic matter, posed by increased forestry and agricultural activity. Although various soil supplements have been considered to enrich P retention in peat soil, there is still a need to develop new ones.

Due to their unique properties, natural zeolites have been used as soil amendments to improve the physicochemical properties of soils (especially for sandy soils), to enrich the retention of plant nutrients in the soil but also in the remediation of soils contaminated by a variety of toxic metals such as Ni, Cd, Pb, Zn, and Co^{1,2}.

Taking into account that FeCLI efficiently adsorbs phosphate from water solution³, this study aimed to examine the influence of FeCLI on P retention in pure peat soil and in lime-enriched peat soil as well as the influence of FeCLI on peat's acidity.

EXPERIMENTAL

Peat soil samples were collected from the top layer (0–20 cm) at various points in the field, which had been treated with different amounts of lime. Prior to the experiments, the samples were air-dried and homogenized at room temperature, sieved through a 2 mm sieve mesh, and stored in polyethylene bags. Samples were labeled as P0 – pure peat soil, P1 and P2, which are samples with different amounts of lime, which was used to raise the pH at a specific value. Ca(OH)₂ was used as the lime source.

The zeolitic tuff containing about 73 wt.% of clinoptilolite (CLI) was obtained from the Zlatokop deposit (Vranjska Banja, Serbia). Primarily, CLI was sieved to obtain particles of 0.063–0.125 mm, washed using deionized water several times and dried overnight at 105 °C until a constant mass. The cation exchange capacity (CEC) measured by the ion exchange with NH₄CH₃COO solution was determined to be 160 cmol/kg.

CLI was converted to FeCLI in accordance with the previously published procedure³. In short, CLI was treated with a $\text{Fe}_2(\text{SO}_4)_3$ solution in an acetate buffer (pH= 3.6). NaOH was added to raise the pH to 7, and the separated solid was heated to a constant mass at 90 °C.

Soil samples characterization: Standard procedures determined the soil samples' physicochemical properties. Soil pH was measured at a soil:water ratio 1:2.5 using a glass electrode pH meter. The organic matter (OM) content was determined as the loss on ignition at 550 °C. The concentration of exchangeable cations (Na, K, Mg, and Ca) was determined after treating the samples with 1 M $\text{NH}_4\text{CH}_3\text{COO}$ solution at pH=7.0 using an atomic absorption spectrophotometer. Exchangeable H^+ was determined by back titration back to pH= 7.00 by use of 0.05 M NaOH.

Zeolite characterization: Both CLI and FeCLI were characterized in detail using various instrumental techniques, and data were described in the previous report³.

Phosphorus adsorption experiments: Adsorption experiments were performed using the batch equilibrium method. Different amounts of peat soil ranged from 20 to 800 mg or a mixture of peat soil and FeCLI (10 mg) was weighed and placed into 50 cm^3 centrifuge bottles. Then, 20 cm^3 of a 2.00 mg dm^{-3} P solution containing 0.0025 mol dm^{-3} CaCl_2 solution (which serves to adjust ionic strength) was added to each bottle. Three drops of toluene were added to each tube to inhibit microbial activity. The suspensions were shaken in a temperature-controlled orbital shaker (125 rpm) at room $25 \pm 1^\circ\text{C}$ for 24 h to reach an equilibrium. Then, the suspensions were centrifuged (8,000 rpm for 10 minutes) and filtered through 0.45 μm filter. The P concentration was determined colorimetrically using the molybdenum–blue method. All adsorption tests were done in triplicate.

Acidity measurements: The change in soil acidity was also studied by measuring the pH value of the soil mixture after the adsorption test. The average pH value was calculated and compared.

RESULTS AND DISCUSSION

Soil characterization: Selected physicochemical properties of the used peat samples are given in Table 1.

Table 1. Selected physicochemical properties of the studied soil samples.

Property	Sample		
	P0	P1	P2
pH	3.58	4.79	6.49
Base saturation, %	6.4	48.9	86.0
Loss on ignition, %	98.4		
Exchangeable Na, cmol_c/kg	0.31	0.32	0.39
Exchangeable K, cmol_c/kg	0.18	0.19	0.20
Exchangeable Mg, cmol_c/kg	5.20	6.20	7.13
Exchangeable Ca, cmol_c/kg	3.31	57.95	109.97
Exchangeable H, cmol_c/kg	124.8	66.4	18.7
Total acidity, meq/g organic matter	3.65	2.45	1.53

As expected, an increase in pH and lime rate is evident. It increased from 3.58 to 4.79 (P1) and 6.49 (P2), most likely due to the precipitation of exchangeable Al and Fe and the deacidification of H^+ by the carbonate ion in the lime. Consequently, the total acidity of peat was decreased with the addition of lime (from 3.65 to 2.45 and 1.53 meq g^{-1} organic matter for P1 and P2, respectively). As a result of liming, the concentration of exchangeable Ca was

significantly increased, from 3.31 to 57.95 and to 109.97 $\text{cmol}_{\text{c}+} \text{kg}^{-1}$ for P1 and P2, respectively. Additionally, there was no notable difference in the exchangeable Na and K concentration before and after liming, whereas the concentration of exchangeable Mg was increased by lime addition, which agreed with the reported findings.

Zeolite characterization: The previous report provided a detailed characterization of the zeolitic tuff both before and after treatment with Fe(III) ³. Conversion of CLI to FeCLI resulted in a) an enrichment in Fe(III) (17 wt.% compared to 0.21 wt. % for CLI), b) an increase in specific surface area from 24 to 140 $\text{m}^2 \text{g}^{-1}$ due to the formation of amorphous Fe(hydro)oxide coverage onto the CLI surface which was confirmed by TEM analysis³.

Phosphorus adsorption in peat soil in the presence of lime and FeCLI

The obtained adsorption results are given in Figure 1. Increasing the amount of peat up to 800 mg increased the P concentration in the soil solution for both pure peat (P0) and peat with lime (P1 and P2). This can be attributed to increased soil organic matter (SOM). SOM affects P adsorption/desorption in the soil through various pathways, and is therefore one of the crucial parameters. SOM favors P release in soil solution by inhibiting P adsorption through competitive adsorption on metal oxides like Fe/Al oxides. In addition, SOM can raise the P concentration in solution by forming more soluble organophosphate complexes, replacing H_2PO_4^- by organic anion on adsorption sites, and increasing the amount of organic P mineralized to inorganic P.

The addition of lime (P1) raised the P concentration in the solution, and subsequent additions caused the P concentration in the soil solution to decrease (P2) (Figure 1). This can be explained by the fact that liming raises the pH of the soil, which encourages P to be released from the surfaces of Al- and Fe(hydro) oxides⁴. However, adding more lime leads to overliming, where P precipitates as insoluble Ca-phosphates.

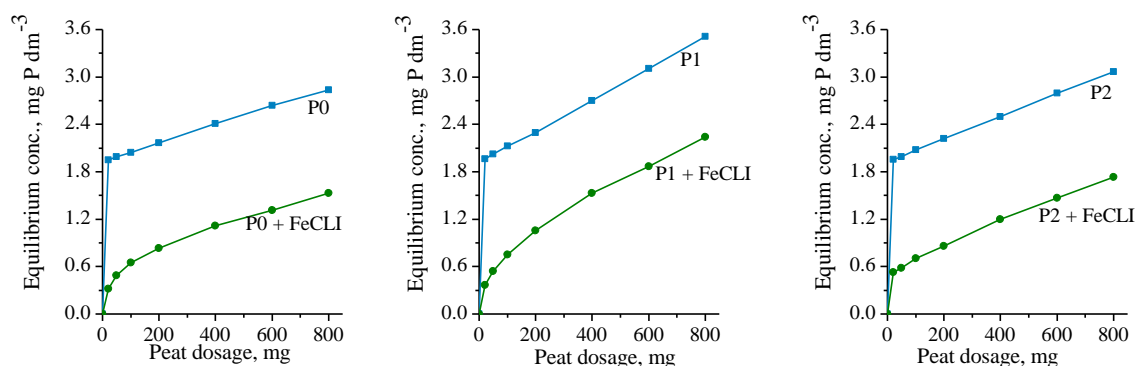


Figure 1. Equilibrium P concentrations for pure peat and amended peat.

The findings indicated that FeCLI influences P adsorption, resulting in a drop in P concentration in the soil solution (Figure 1). Both pure peat and peat that had been amended with lime showed the effect. Furthermore, the effect is visible for every studied peat amount. This can be attributed to a high affinity of FeCLI toward P. FeCLI's role in P retention may also be explained by SOM's ability to complex iron present on FeCLI, creating $\text{FeCLI-PO}_4\text{-SOM}$ complexes that increase the number of P adsorption sites.

The P concentration in the soil solution increased as the amount of peat increased, indicating competition between SOM and P functional groups for more adsorption sites on FeCLI. The curved line at low peat doses, especially for P0 and P1 in Figure 1, indicates this. FeCLI lowers the P concentration in the solution by approximately 84% (P0), 70% (P1), and 73% (P2) for 20 mg of peat. The P concentration in the solution decreased by 46% (P0), 36%

(P1), and 43% (P2) by using 800 mg of peat, indicating the significance of the FeCLI amount added in peat.

Acidity measurements

Figure 2 displays the findings from measuring the pH of the soil solutions following the adsorption tests. As expected, a higher peat content causes the pH to drop: in P0, from 4.06 to 3.12; in P1, from 4.94 to 4.31; and in P2, from 6.17 to 5.84. Up to 200 mg of peat, the pH decreased more noticeably before slowing down. Many acid functional groups, including carboxylic and phenolic ones, are present in SOM, allowing H⁺ ions to dissociate and lower pH. The presence of FeCLI raised the pH of the solution, indicating that its addition can lessen soil acidity. This may be explained by the decrease of SOMs due to their adsorption onto FeCLI. However, as SOM levels rise, the effect becomes less pronounced.

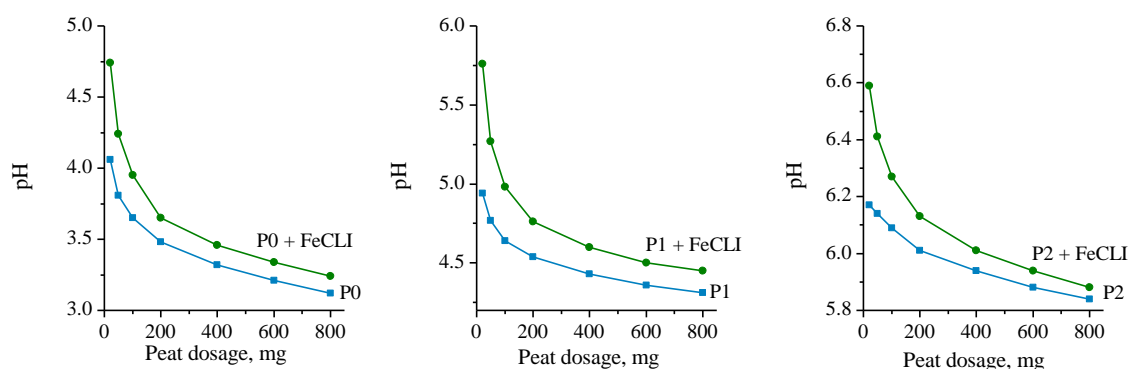


Figure 2. The effect of FeCLI presence on soil pH.

CONCLUSION

The presented results suggest that FeCLI can be a beneficial soil supplement supporting agricultural practices and water conservation. FeCLI enhances the P retention in both pure peat soil and peat that has been lime-amended. With more peat present, the supplement's impact has been less apparent, with a reduction in P concentration in the range of 46% to 84%. The reduction in P concentration in the lime-amended peat ranged from 36% to 73%. Both pure peat and lime-enriched peat have a lower acidity when FeCLI is present.

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