E! 4208 PUREWATER

Prirodni zeoliti u sustavu kvalitete voda

Natural zeolites in water quality system

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Sveučilište u Zagrebu
Tekstilno-tehnološki fakultet
Zavod za primijenjenu kemiju
Laboratorij za analitičku kemiju

Radionica: FP7 - Program jačanja istraživanja i razvoja za napredne poduzetnike
Županijska komora Karlovac, 8. prosinca 2011.

Eureka program

- EUREKA je europska mreža za tržišno usmjereno istraživanje i razvoj i pomaže izgradnji međunarodnog partnerstva u cilju jačanja europske konkurentnosti.
- Očekivani rezultati projekata su novi proizvodi ili usluge koji promiču napredne tehnologije i imaju svoje mjesto na tržištu.
- Članice inicijative su zemlje koje su odlučile dati podršku međunarodnim projektima iz nacionalnih proračuna.
- Danas EUREKA okuplja i povezuje tvrtke i istraživačke institucije iz 40 zemalja te Europske komisije, koja ima status punopravne članice.

Ciljevi

- Primarni cilj programa EUREKA je potaknuti tvrtke na ulaganje u aktivnosti istraživanja i razvoja te na taj način jačati njihov inovacijski kapacitet.

- EUREKA pomaže tvrtkama, malim i srednje velikim poduzetnicima da ujedine svoje resurse, da putem međunarodne suradnje i suradnje sa znanstveno istraživačkim organizacijama razviju inovativne proizvode, procese ili usluge te ujedno stvore pretpostavke za međunarodni tržišni plasman.

Kriteriji za rangiranje i prihvaćanje projekata

- sastoje se od osnovnih i posebnih kriterija.
  - Osnovni kriteriji definirani su Hannoverskom deklaracijom, temeljnim osnivačkim dokumentom inicijative, dok
    - Temelj za utvrđivanje posebnih kriterija su odrednice strategija znanstvenog, tehnološkog i gospodarskog razvitka Republike Hrvatske.Pretprijava projekta vrši se slanjem ispunjenog obrasca EUREKA Project Form (ili Application form). Nacionalnom koordinatoru projekata (NPC)
  - posebne kriterije utvrđuju Ministarstvo znanosti, obrazovanja i športa i Poslovno-inovacijski centar Hrvatske BICRO.).Na temelju Pretprijave projekt se interno ocjenjuje prema osnovnim kriterijima:

Kriteriji za rangiranje i prihvaćanje projekata

- partneri moraju biti iz najmanje dvije različite zemlje sa statusom punopravnog članstva
- projekt mora imati primjenu u civilnom sektoru
- projekt mora imati jasno izraženu tehnološku inovativnost
- projekt mora biti ekološki podoban

Kriteriji za rangiranje i prihvaćanje projekata

Posebni kriteriji za Hrvatsku:

- projekt se uklapa u nacionalne prioritete gospodarskog i tehnološkog razvoja
- projekt omogućuje proizvodnju u Republici Hrvatskoj
- projekt omogućuje ulaganja u Republiku Hrvatsku
- projekt omogućuje transfer tehnologije
- projekt otvara pristup vanjskim tržištima hrvatskom partneru
- projekt otvara nova radna mjesta
- projekt omogućuje značajnu referenciju hrvatskom partneru

- Kod navedenih posebnih kriterija procjenjuje se:
  - Kvaliteta menadžmenta
  - Stupanj inovativnosti
  - Komercijalni potencijal
  - Konkurentska prednost

Natjecatelji i korisnici

- Program je namijenjen malim i srednje velikim trgovačkim društvima.
- Znanstveno-istraživački partneri mogu se uključiti kao dodatni partneri poduzećima i ne mogu prijaviti projekt samostalno bez suradnje s poduzećima.
- Mogući znanstveno-istraživački partneri su javni i privatni znanstveno-istraživački instituti te Sveučilišta, veleučilišta i druge visokoškolske ustanove.
- EUREKA projekti su međunarodnog karaktera.
- Projektni partneri moraju biti iz najmanje dvije različite zemlje sa statusom punopravnog članstva.
- Popis zemalja članica :
  - Austria (1985), Belgium (1985), Croatia (2000), Cyprus (2002), Czech Republic (1995), Denmark (1985), Estonia (2001), Finland (1985), former Yugoslav Republic of Macedonia (2008), France (1985), Germany (1985), Greece (1985), Hungary (1992), Iceland (1985), Ireland (1985), Israel (2000), Italy (1985), Latvia (2000), Lithuania (1999), Luxembourg (1985), Malta (2006), Monaco (2005), the Netherlands (1985), Norway (1985), Poland (1995), Portugal (1985), Romania (1997), Russian Federation (1993), San Marino (2005), Serbia (2002), Slovak Republic (2001), Slovenia (1994), Spain (1985), Sweden (1985), Switzerland(1985), Turkey (1985), Ukraine (2006), United Kingdom (1985), European Commission (1985).
  - EUREKA NIPs: Albania, Bosnia and Herzegovina,Bulgaria.
  - EUREKA Associated country: South Korea
- BICRO (Poslovno-inovacijski centar Hrvatske) nudi podršku pri pronalaženju stranih partnera putem Europske poduzetničke mreže EEN, u kojoj je BICRO glavni partner u Hrvatskoj za inovacije te transfer tehnologije i znanja.

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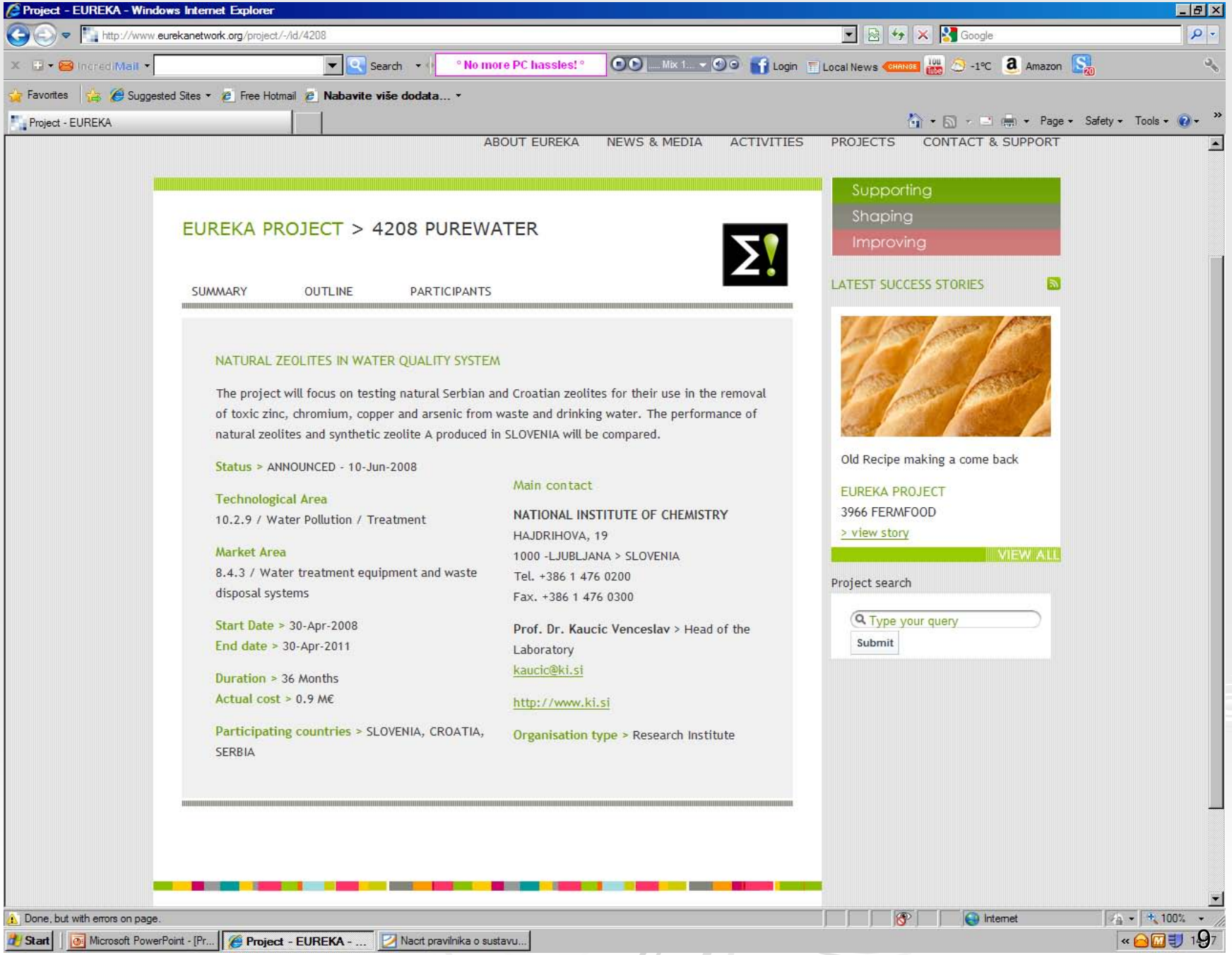
Natural zeolites in water quality system

- Koordinator: National Institute of Chemistry Ljubljana, Slovenia Prof. Venčeslav Kaučič
- Silkem d.o.o.– partner iz industrije

- Fakultet kemijskog inženjerstva i tehnologije
- Tekstilno-Tehnološki fakultet
- CWG d.o.o. – partner iz industrije

- Tehnološko metalurški fakultet
- Poljoprivredni fakultet
- GM water d.o.o – partner iz industrije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NATURAL ZEOLITES IN WATER QUALITY SYSTEM
The project will focus on testing natural Serbian and Croatian zeolites for their use in the removal of toxic zinc, chromium, copper and arsenic from waste and drinking water. The performance of natural zeolites and synthetic zeolite A produced in SLOVENIA will be compared.
Status > ANNOUNCED - 10-Jun-2008
Technological Area
10.2.9 / Water Pollution / Treatment
Market Area
8.4.3 / Water treatment equipment and waste disposal systems
Start Date > 30-Apr-2008
End date > 30-Apr-2011
Duration > 36 Months
Actual cost > 0.9 M€
Participating countries > SLOVENIA, CROATIA, SERBIA
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HAJDRIHOVA, 19
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Povijest prirodnih zeolita

- 1756. godine otkriće stilbita - prvog prirodnog zeolita - švedski mineralog Axel Fredrik Cronstedt
- Nazvao ga je zeolit, što dolazi od grčke riječi *zeo* i *lithos* sa značenjem “kamenje koje vrije” – zbog vizualnog efekta primijećenog pri zagrijavanju zeolita

Prirodni zeoliti danas

- Zeoliti čine više od 90 % mnogih sedimentnih stijena vulkanskog porijekla i široko su rasprostaranjeni po cijelom svijetu
- Poznato je gotovo 50 zeolitnih materijala
- Samo 6 ih se nalazi u znatnim količinama
  - Habazit
  - Klinoptilolit
  - Mordenit
  - Erionit
  - Hojlandit
  - Filipsit

Klinoptilolit $(Na,K)_6Si_{30}Al_6O_{72} \cdot 24\ H_2O$

- Najrasprostranjeniji prirodni zeolit
- Klinoptilolitom bogate stijene sadrže 60-90 % klinoptilolita uz feldspate, gline i kvarc kao mineralne nečistoće
- Glavna nalazišta klinoptilolitom bogatih stijena rasprostranjena su cijelim svijetom (Argentina, Kuba, Meksiko i SAD) posebno u Europi, odnosno u istočnoj Europi (Bugarska, Grčka, Hrvatska, Madžarska, Rumunjska, Slovačka, Srbija, Turska)
- Hrvatska: nalazište u kamenolomu Donje Jesenje (Maceljska gora); udjel klinoptilolita 50-60 %

Proizvodnja prirodnih zeolita

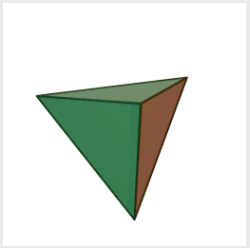
- Iskorištavanje je vrlo jednostavno

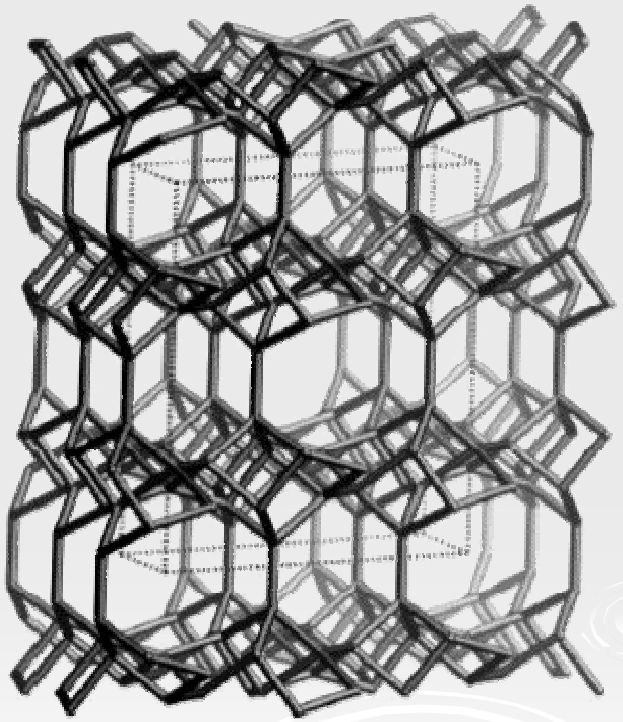


- Prije 25 godina: oko 300 000 t
- Danas: 4 000 000 t/god
- Povećanje proizvodnje: 10% /god
- Cijena: 50-300 USD/t

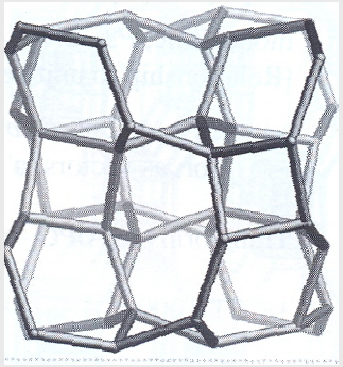
Struktura i svojstva prirodnih zeolita

- Zeoliti su hidratizirani alumosilikati, jedinstvene trodimenzionelne strukture, sastavljene od SiO_4^{4-} i AlO_4^{5-} tetraedara spojenih kisikovim atomima,
- specifične su mrežaste strukture sa šupljina međusobno povezanim kanalima određenog oblika i veličine

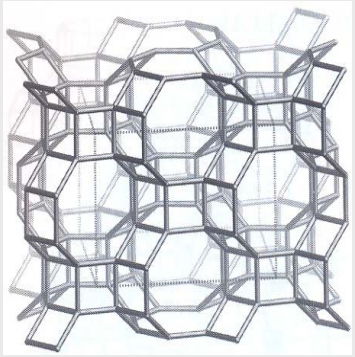




TRODIMENZIONALNA STRUKTURA ZEOLITA



analcim



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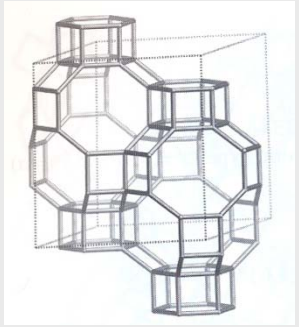

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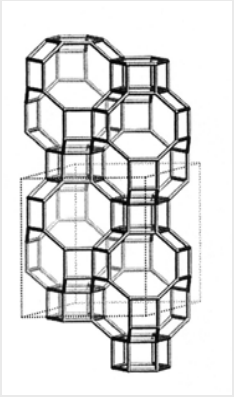

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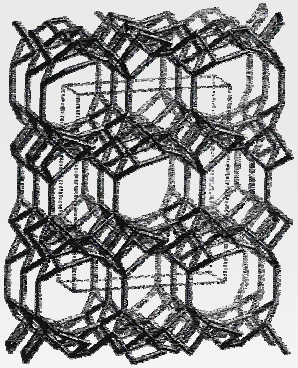
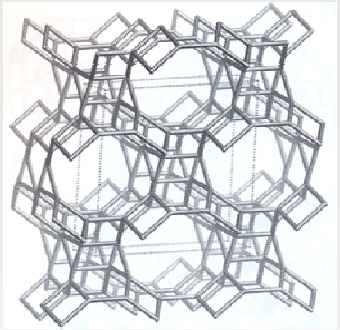


klinoptilolit



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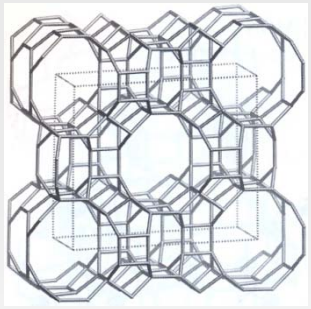


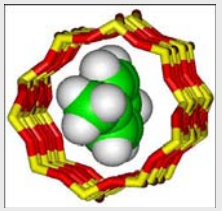
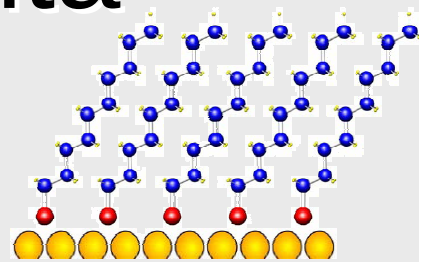
mordenit

Karakterizacija prirodnih zeolita

- Kemijska analiza
- XRD - metoda difrakcije rendgenskih zraka na praškastom uzorku
- EDS – elementarna analiza pomoću disperzijske spektroskopije rendgenskih zraka
- XANES/EXAFS
- NMR, IR
- SEM – skenirajući elektronski mikrosokp
- TG/DTG – termogravimetrija/derivativna termogravimetrija
- DSC – diferencijala skenirajuća kalorimetrija
- TEM -

Primjena prirodnih zeolita

- životinjska higijena,
- životinjska ishrana,
- građevinski materijali,
- gnojiva,
- Farmaceutski pripravci
- Nosači za katalizu, mikoorganizme
- sorbensi mirisa i plinova,
- kao membrane i filteri za pročišćavanje voda, gradskih i industrijskih otpadnih voda,
- za uklanjanje radionuklida iz voda nuklearnih postrojenja






Prednosti prirodnih zeolita

- Široka rasprostranjenost u prirodi
- Niska cijena eksploatacije
- Visoka učinkovitost uklanjanja zagađivala iz voda procesima ionske izmjene i/ili adsorpcije
- Visoka učinkovitost uklanjanja iona metala iz voda, otpadnih voda i vodnih sustava
- Zamijenjivi kationi nisu toksični (Na, K, Ca, Mg)
- Jednostavno zbrinjavanje zeolita zasićeng teškim metalima nakon ionske izmjene (dodatak cementnim kompozitima, betonima, u izgradnji cesta...)
- Učinkoviti su i pri uklanjanju štetnih aniona (arsenati, kromati itd.) nakon modifikacije – funkcionalizacije površine prirodnog zeolita

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Project Description

Many toxic heavy metal ions are discharged into the environment with industrial wastes causing serious water and soil contamination. Heavy metal ions such as Cr3+, Cu2+, Zn2+ and Pb2+ are common metal ions, which accumulate in living organisms causing numerous disorders and diseases. They are also common groundwater contaminants at industrial installations. Arsenic is a well known metalloid which causes serious problems in drinking water since it is extremely poisonous. The presence of arsenic in water is due to the dissolution of minerals from subterranean strata or from an anthropogenic origin such as the leaching of arsenic compounds from smelting of metal ores, agricultural pesticides or wood preservatives. Arsenic contamination of groundwater as a source of drinking water has been a health risk for many regions around the world. The WHO (World Health Organisation) established that even a low concentration of arsenic in drinking water produces negative effects on human health, therefore the arsenic reference value for drinking water is 0.010 mg/dm3. Many wells spread all over the world exceed this value. Thus, there is still an urgent need for development of a cost-effective method for arsenic removal from groundwater and wastewater. Numerous methods exist for removing heavy metal and arsenic ions from water. Precipitation, ultra-filtration, reverse osmosis, ion exchange and adsorption are some of the commonly used techniques. As it is easy to remove the sorbent from aqueous media after treatment, sorption is generally considered to be the most suitable method. The use of alternative low-cost materials such as potential sorbents has recently been extensively studied. The processes using activated carbons or alumina as adsorbent are considered to be particularly competitive and effective, however they are not suitable in developing countries due to the high costs associated with the production and regeneration of the spent adsorbent. Accordingly, ion exchange is considered to be cost-effective only when low-cost exchangers such as natural zeolites are used. Zeolites are naturally occurring aluminosilicates. They are well known for their ion exchange, catalytic and sorption properties. The structures of zeolites consist of three-dimensional networks of SiO4 and AlO4 tetrahedra arranged in an alternating manner. This produces negatively charged lattice, where the presence of exchangeable captions such as sodium,

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different sorption and ion-exchange behaviour, the difference being mainly caused by a different composition of zeolitic tuffs. In this project, two large clinoptilolite deposits from SERBIA (Vranjska Banja and Beocin) and one from CROATIA (Donje Jesenje) will be investigated for use in heavy metal and arsenic ion removal from waste and drinking water. Ion exchange and adsorption methods will be used for the pollutant removal. The performance of natural zeolitic tuffs will be compared with the adsorption characteristics of synthetic zeolite A (4 nm channels) produced in SLOVENIA in order to evaluate their technological and economical advantages and disadvantages.
Technological Development Envisaged
In water management processes, water as a raw material should be optimally utilised. The advantage of zeolites is their ion exchange properties, which enable the return of the purified water back into the production processes. The concentrated solutions obtained as a result of ion exchanger regeneration processes can also be returned into production processes as a raw material. Metal Cr and As species are present in water solutions as different aqua complexes. The ionic radii of the investigated cations are smaller than the radii of the CLI channels, so that all the cations can pass readily through the channels. This means that the ion exchange process will take place readily and without hindrance. However, the literature reports have shown that both the ion exchange and the chemisorption act toward the immobilisation of heavy metal cations in natural zeolites. Since the ion exchange is reversible and chemisorption is an irreversible process - resulting in the formation of stable complexes between the zeolite lattice and heavy metal ions - detailed structural investigations of the metal-containing CLI will be performed. The efficiency of the As removal by zeolite is greatly affected by the presence of iron ions. Also, surfactant-modified clays show a strong affinity to anionic species. Both methods will be studied for the immobilisation of arsenic species on the CLI samples and the As removal from drinking water.
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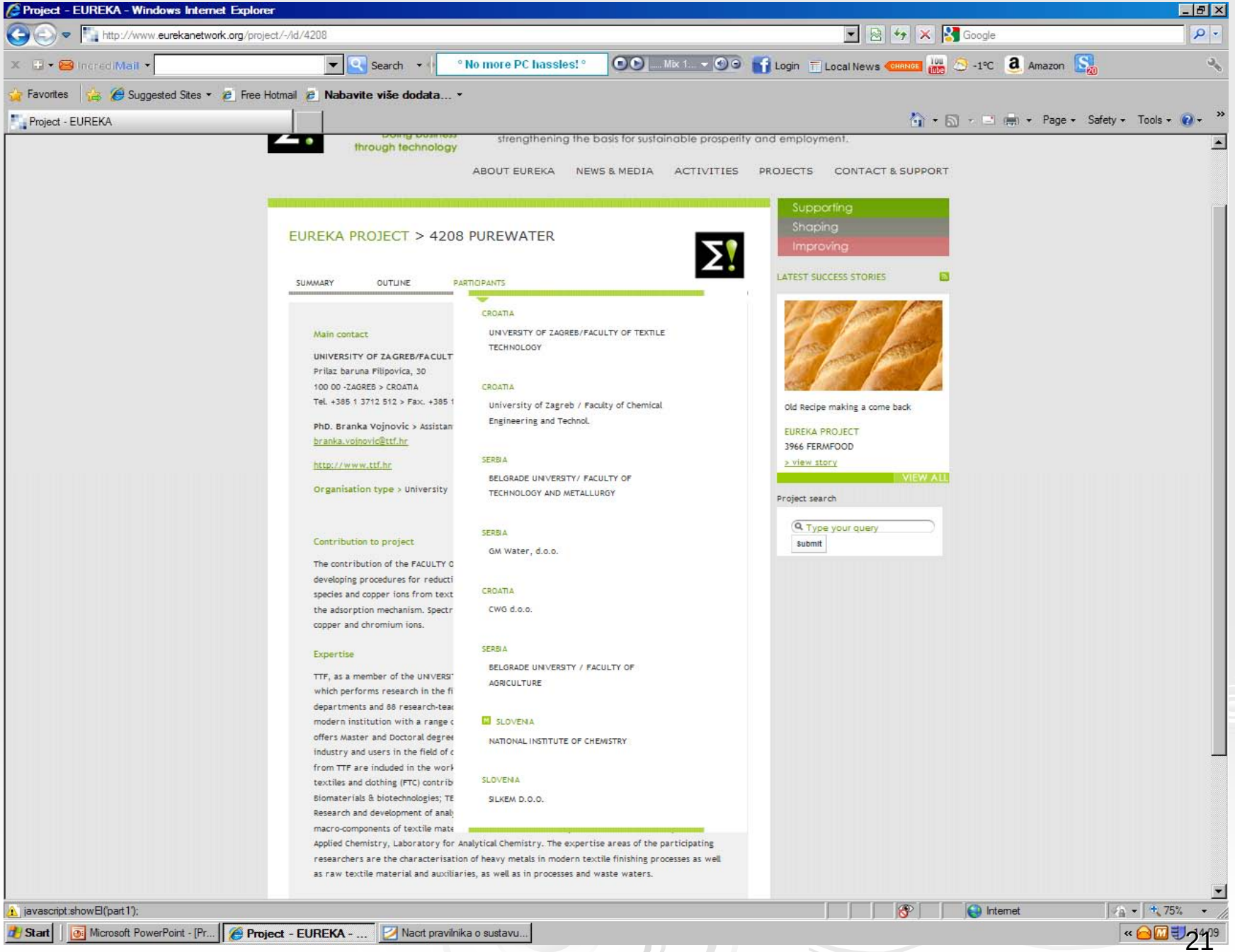
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University of Zagreb / Faculty of Chemical Engineering and Technol.
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BELGRADE UNIVERSITY/ FACULTY OF TECHNOLOGY AND METALLURGY
SERBIA
GM Water, d.o.o.
CROATIA
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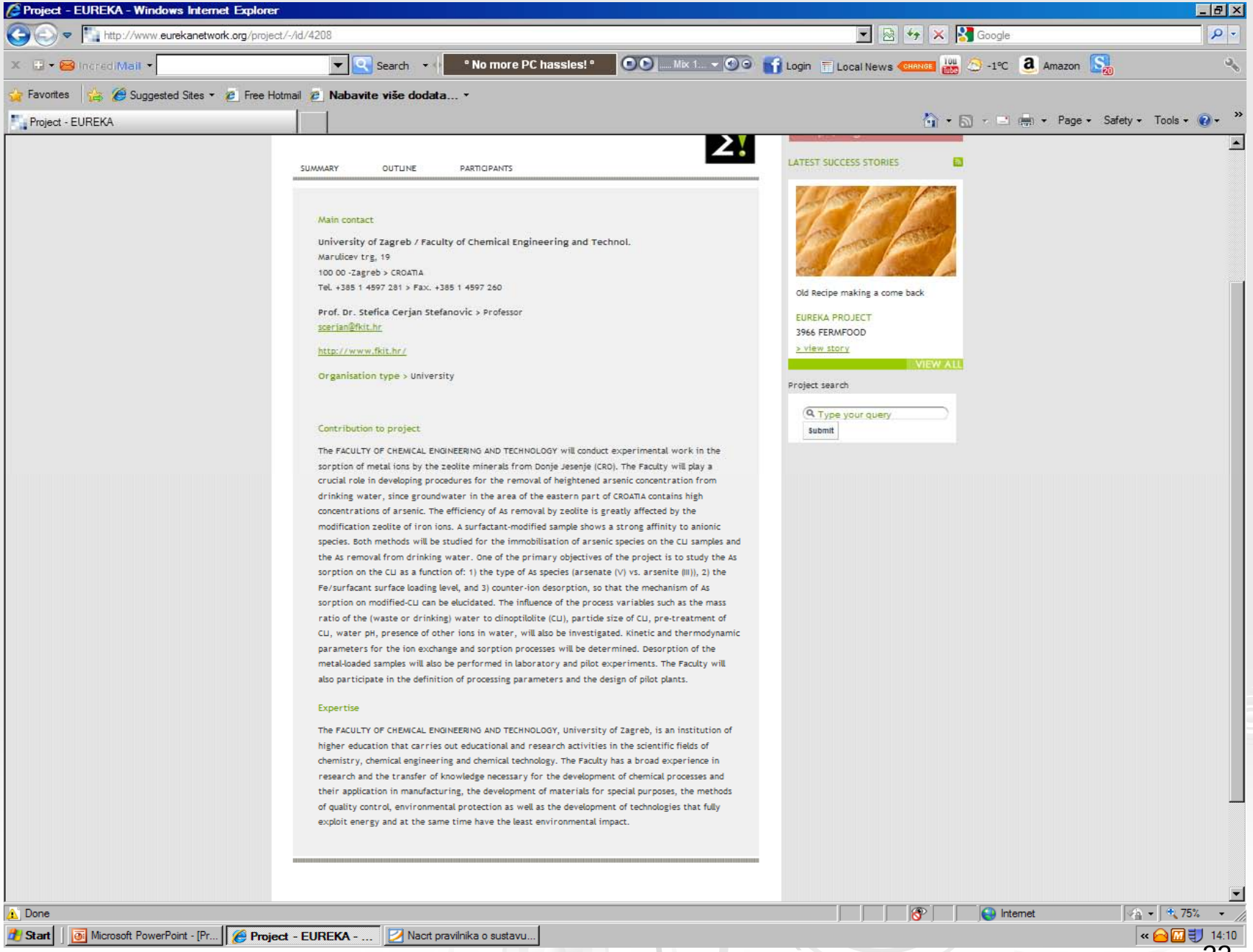
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Organisation type > University
Contribution to project
The FACULTY OF CHEMICAL ENGINEERING AND TECHNOLOGY will conduct experimental work in the sorption of metal ions by the zeolite minerals from Donje Jesenje (CRO). The Faculty will play a crucial role in developing procedures for the removal of heightened arsenic concentration from drinking water, since groundwater in the area of the eastern part of CROATIA contains high concentrations of arsenic. The efficiency of As removal by zeolite is greatly affected by the modification zeolite of iron ions. A surfactant-modified sample shows a strong affinity to anionic species. Both methods will be studied for the immobilisation of arsenic species on the CLI samples and the As removal from drinking water. One of the primary objectives of the project is to study the As sorption on the CLI as a function of: 1) the type of As species (arsenate (V) vs. arsenite (III)), 2) the Fe/surfacant surface loading level, and 3) counter-ion desorption, so that the mechanism of As sorption on modified-CLI can be elucidated. The influence of the process variables such as the mass ratio of the (waste or drinking) water to clinoptilolite (CLI), particle size of CLI, pre-treatment of CLI, water pH, presence of other ions in water, will also be investigated. Kinetic and thermodynamic parameters for the ion exchange and sorption processes will be determined. Desorption of the metal-loaded samples will also be performed in laboratory and pilot experiments. The Faculty will also participate in the definition of processing parameters and the design of pilot plants.
Expertise
The FACULTY OF CHEMICAL ENGINEERING AND TECHNOLOGY, University of Zagreb, is an institution of higher education that carries out educational and research activities in the scientific fields of chemistry, chemical engineering and chemical technology. The Faculty has a broad experience in research and the transfer of knowledge necessary for the development of chemical processes and their application in manufacturing, the development of materials for special purposes, the methods of quality control, environmental protection as well as the development of technologies that fully exploit energy and at the same time have the least environmental impact.
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SUMMARY OUTLINE PARTICIPANTS

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UNIVERSITY OF ZAGREB/FACULTY OF TEXTILE TECHNOLOGY
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Organisation type > University

Contribution to project

The contribution of the FACULTY OF TEXTILE TECHNOLOGY (TTF) will be in experimental work in the developing procedures for reduction of coloured textile waste waters and eliminating of chromium species and copper ions from textile effluents after treatment by natural zeolites through studying the adsorption mechanism. Spectroscopic methods will be suggested for measuring the content of copper and chromium ions.

Expertise

TTF, as a member of the UNIVERSITY OF ZAGREB, is the only institution in the REPUBLIC OF CROATIA which performs research in the field of textile science and technology. It consists of seven departments and 88 research-teaching staff supervising 1000 students' at all educational levels. It is a modern institution with a range of education levels in accordance with the Bologna process and offers Master and Doctoral degrees, as well as scientific research and technical scientific services for industry and users in the field of chemical, mechanical, and clothing technology and design. Experts from TTF are included in the work of the European Technology Platform (ETP): for the future of textiles and clothing (FTC) contributing in sectors: TEG 2: Functionalisation of textiles; TEG 3: Biomaterials & biotechnologies; TEG 4: New textile products;

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EUREKA PROJECT > 4208 PUREWATER

SUMMARY OUTLINE PARTICIPANTS

Main contact

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Dipl. Ing. Mario Skrapec > General manager
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http://www.cwg.hr

Organisation type > Governm./Nat. Admin.

Contribution to project

Regarding the wide range of new technology used in water treatment, CWG's contribution to this project will be all the equipment needed for quality and fruitful results. Along with high-technology equipment, their service field team is fully equipped for any task. CWG D.O.O. will support the project team with their know-how experience in water treatment along with the use of the best supporting materials and best units to achieve excellent results in this project. CWG D.O.O. will play a crucial role in the technological part of the project where the knowledge will be transferred into practice, i.e. the zeolites with the best adsorption-desorption characteristics will be used in the new water purification systems.

Expertise

CWG D.O.O.'s core business and expertise stem from 25 years of work on water treatment processes, technology and wastewater. CWG has always been very forward-looking and has educated the industrial sector about reaching higher levels of environment-friendly equipment and waste reuse. From our experience and the quality indicators in the industrial sector over the years, CWG has built up a solid knowledge base. Our major goal is to reach a maximum exploitation level and minimise production costs. Based on CWG®s experiences and current know-how in the water

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