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Novel NO_x Storage Materials via Well-Controlled Surface Chemistry of Oxide Nano-domains

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NO_x Storage Reduction Catalyst (NSR) technology is recently developed by Toyota Motor Co. in order to convert hazardous pollutant gases such as NO_x, CO_x and NO_x that are originating from diesel-engine based automotive operations. Although this revolutionary technology is widely used commercially in Japan, its world-wide commercialization has not been achieved due to various technical limitations related to the intricate NSR catalysis. In this talk, I will present examples regarding our recent studies on NSR catalysts at Bilkent University Chemistry Department, which focus on the development of novel complex mixed-oxide materials whose surface structure is controlled at the nanometer scale in order to improve their NO_x storage properties. Synthesized novel materials were thoroughly characterized with a variety of spectroscopic, diffraction and imaging techniques such as XRD, BET, Raman Spectroscopy, SEM, EDX, TEM, EELS, XPS, FTIR and TPD. NO_x uptake mechanisms of these novel mixed-oxide systems were also investigated using a dedicated in-situ FTIR-TPD reactor setup. In the light of these synthesis, characterization and NO_x storage experiments, new valuable “*structure-function*” relationships were unveiled regarding the behavior of NSR catalysts.

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submitted.

Effect of catalyst preparation conditions on activity and selectivity of PtSn/SiO₂ catalyst in citral hydrogenation

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Abstract- In this study, the citral hydrogenation reaction in liquid phase over silica gel supported Pt and PtSn catalysts were studied. It is desired to hydrogenate carbonyl group (C=O) of citral selectively to produce valuable unsaturated alcohols, namely nerol and geraniol. Catalysts were prepared by impregnation, co-impregnation and catalytic reduction methods. The effects of parameters investigated in the present study included catalyst activation temperature (350 °C and 450 °C), calcination temperatures (400 °C and 500 °C), and catalyst activation without calcination. Pt loading was 1 wt% and Sn loading was 0.3 wt%. The highest selectivity to citronellol and geraniol (S_{COL+GE}) was 39.0 % over Pt/SiO₂. Bimetallic catalysts, PtSn/SiO₂, prepared by co-impregnation and catalytic reduction methods gave S_{COL+GE} of 16.4 % and 9.0 %, respectively. Increasing calcination and reduction temperatures of bimetallic catalysts increased S_{COL+GE} .

Catalysts are compounds which are nanoparticles, speed up or slow down chemical reactions without being consumed by them. The chemical and drug industries spend lots of money for catalysts that are needed to process drugs and other high-value chemicals. In between catalysts, nanoparticles combining platinum and other metals such as tin are prized for reactions including hydrogen because of pairing atoms of hydrogen platinum and other metals are good at sticking these pairs and leaving the particular hydrogen atoms available for reactions with other molecules [1].

Citral is an important component of the lemongrass oil. There are three hydrogenation sites of citral, isolated C=C double bond, conjugated C=C double bond and a carbonyl group (C=O). Citral presents as cis- and trans- isomers. Unsaturated alcohols such as nerol and geraniol are the most important products of the citral hydrogenation reaction. These products are used in pharmaceuticals, perfumes and flavours [2].

Unsaturated alcohols are produced by the selective hydrogenation. The selective hydrogenation of citral over metal catalysts provides saving chemicals and reducing wastes. In the previous studies, it was mentioned that the reaction selectivity was affected by particle sizes, support material and metals (Sn, Ge, Fe and Pt). The silica and alumina are used as support material for the monometallic Pt catalysts [3].

The effects of parameters investigated in the present study included catalyst activation temperature (350 °C and 450 °C), calcination temperatures (400 °C and 500 °C), the solvent type (ethanol, 2-pentanol), and catalyst activation without calcinations. Pt/SiO₂ was prepared by impregnation (I) method while PtSn/SiO₂ by co-impregnation (CI), successive impregnation and catalytic reduction (CR) methods were applied.

For the preparation of 1 % Pt loaded Pt/SiO₂, Pt source, calcined silica gel (at 500 °C, 6 h) and pure water were mixed. The water was evaporated at 60 °C under 150 mbar vacuum in rotary evaporator for 3 hours. The Pt/SiO₂ catalyst was dried at 110 °C overnight and calcined at 500 °C for 6 hours under 70 ml/min flowing dry air. Bimetallic catalysts having 0.3 wt% tin with Sn/Pt molar ratio of 0.5 were prepared. In the catalytic reduction method [3] for PtSn/SiO₂, the first step was just like as impregnation method. The catalyst was activated at the activation temperatures under H₂ flow for 4h. Then it was placed in a glass reactor and tetrabutyl tin dissolved in toluene was added. The slurry was mixed for 1 h under H₂ bubbling. After that it was dried overnight. The co-impregnation method for PtSn/SiO₂ was just like as impregnation method but the difference was that Pt and Sn sources were mixed together in toluene. Before the reaction, all of the catalysts were activated

either at 350 °C or 450 °C under 4 bar H₂ gas pressure. Then citral hydrogenation reaction was performed in a semi-batch reactor at 80 °C, 6 bar H₂ gas pressure and 1000 rpm stirring speed [4].

XRD and SEM analyses showed that SiO₂ gel had an amorphous structure. Selectivities to citronellol and geraniol (S_{COL+GE}) obtained over Pt/SiO₂ and PtSn/SiO₂ catalysts are given in Figure 1. GC-MS analysis showed a citral conversion of 73.0 % and selectivity to citronellol and geraniol (S_{COL+GE}) of 39.0 % over Pt/SiO₂. Tin addition affected the metal activity differently for different preparation methods. Bimetallic catalysts PtSn/SiO₂ prepared at the same condition (calcination temperature of 500 °C and reduction temperature of 350 °C) by CI and CR gave a little bit higher conversion, for CI 79.3 % and for CR 76.0 %. However, they gave lower S_{COL+GE} than Pt/SiO₂. It was 16.4 % by CI and 9.0 % by CR method. Increasing the reduction temperature to 450 °C decreased conversion for both CI and CR. The conversions were 70.4 % (S_{COL+GE} , 16.8 %) and 63.2 % (S_{COL+GE} , 14.5 %) for CI and CR, respectively. When the calcination temperature decreased to 400 °C for catalysts prepared by CI method, both conversion (57.0 %) and S_{COL+GE} (9.0 %) decreased.

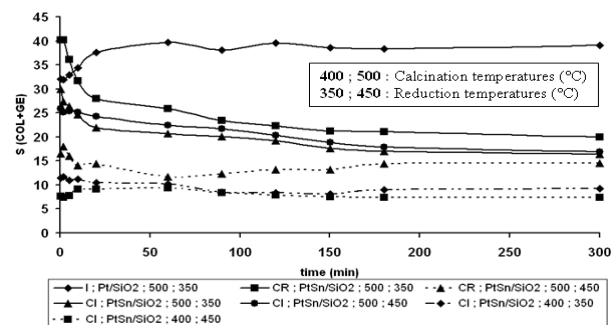


Figure 1. Selectivities to citronellol and geraniol (S_{COL+GE}) obtained over Pt/SiO₂ and PtSn/SiO₂ catalysts

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Anticancer Drug Interaction with DNA Immobilized onto Redox Polymer Coated Electrodes

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Abstract—The interaction of mitomycin C (MC) and DNA immobilized onto poly(vinylferrocenium) (PVF⁺) modified platinum (Pt) and gold (Au) electrodes was examined in this work.

The interaction of anticancer drugs with DNA is an important topic for studies in drug discovery and pharmaceutical development processes [1]. The investigation of interactions of some anticancer agents with DNA was employed by a variety of techniques; such as, DNA-footprinting, nuclear magnetic resonance (NMR), mass spectrometry (MS), spectrophotometric methods, vibrational spectroscopy (Raman and infrared), capillary electrophoresis, surface plasmon resonance (SPR) and electrochemistry [2-4]. Among these techniques, electrochemical techniques received considerable attention [5-7].

Mitomycin C (MC) is an anticancer antibiotic drug which was isolated from *Streptomyces caespitosus* and it is used in clinical chemotherapy against a broad spectrum of solid tumors. Mitomycin C has cytotoxic character and this molecule gives damage to normal human cells [8].

In this work, the interaction of mitomycin C (MC) and DNA immobilized onto poly(vinylferrocenium) (PVF⁺) modified platinum (Pt) and gold (Au) electrodes was investigated. The electrochemical behavior of DNA immobilized polymer modified electrode was examined in the absence/presence of MC by using differential pulse voltammetry (DPV). The magnitude of electroactive DNA base, guanine was monitored before and after interaction between MC and DNA. In order to obtain more sensitive electrochemical signals, the effects of MC concentration and MC interaction time were examined. Alternating current (AC) impedance spectroscopy and scanning electron microscopy (SEM) were also used for the characterization of polymer modified electrodes.

PVF⁺ modified electrode provided an appropriate interface for MC-DNA interaction. Compared with other drug-DNA interaction techniques based on electrochemistry [1,8], these polymer electrodes can provide simple, easy, fast and cheap detection scheme for drug-DNA interaction with a satisfactory detection limit in a linear dynamic range. The detection limit corresponded to 0.47 µg/mL for MC using dsDNA immobilized polymer modified Pt electrode (n = 3). The polymer modified Pt electrode for MC-DNA interaction explored in the study presented a good linearity ($R^2 = 0.992$) to MC in a high concentration range of 25 to 75 µg/mL. The detection limit corresponded to 0.09 µg/mL for MC using dsDNA immobilized polymer modified Au electrode (n = 3). The

polymer modified Au electrode for MC-DNA interaction showed a linearity ($R^2 = 0.973$) to MC in a high concentration range of 10 to 50 µg/mL.

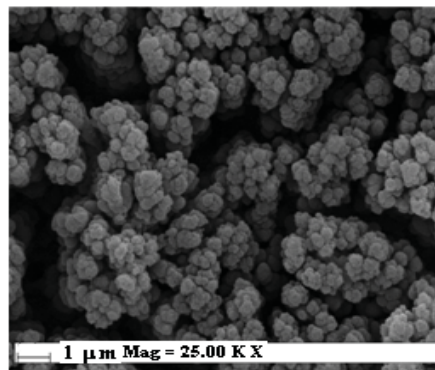


Figure: SEM image of nanostructured polymer

The determination of the interaction of DNA with drugs using polymer modified electrodes can play an important role in the design of new chemotherapeutic compounds and diagnosis techniques. This study was supported by Turkish Scientific and Technological Council (TUBITAK) (Project no: 106T065). A.E. expresses her gratitude to the Turkish Academy of Sciences (TUBA) as an Associate member for their support.

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Catalytic Wet Air Oxidation of Acetic Acid over a Pt/SiO₂ Catalyst at Atmospheric Pressure

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Abstract- Aqueous solutions of acetic acid (AA) were treated by catalytic wet air oxidation (CWAO) using SiO₂ supported Pt catalyst at atmospheric pressure at a temperature of 333 K. The catalyst was characterized by nitrogen adsorption, SEM, IR and XRD measurements. The effects of parameters such as acetic acid initial concentration, loading of catalyst and temperature were investigated on the degradation degree of AA. In the oxidation of acetic acid, intermediates such as oxalic acid and formic acid were formed and acetic acid was not totally oxidized. 25 % of degradation could be achieved for 150 mL of 3000 ppm AA solution in the presence of 1 g catalyst with 3 ml/s air flow rate after a reaction time of 3h. CWAO reaction of AA was described by an empirical rate law with an activation energy of 141.77 kJ/mol.

Catalytic wet air oxidation (CWAO) is a very useful technique for treating wastewaters where the concentrations of pollutants are too low for incineration process or too toxic for biological treatment. During the wet air oxidation of wastewaters containing aromatic compounds, several intermediates including mono and di aliphatic carboxylic acids and unsaturated carboxylic acids are formed and they are quite refractory to chemical oxidation.

Acetic acid (AA) is the most refractory acid among the low molecular weight carboxylic acids such as formic, oxalic, butyric, maleic, succinic and stearic acids. The main sources of emissions of acetic acid are from the industries that produce it or use it in production of polyethylene, terephthalate, cellulose acetate and vinyl acetate. In literature, platinum, ruthenium, palladium and iridium catalysts have been prepared on alumina, carbon, ceria, TiO₂ and ZrO₂ supports and tested in CWAO of acetic acid. High temperatures (473 K) and high oxygen partial pressures (20 bar) are required for CWAO of acetic acid. In this study CWAO of AA aqueous solution was investigated over a Pt/SiO₂ catalyst at atmospheric pressure at a temperature of 333 K [1-2]

In this study, SiO₂ supported Pt catalyst (1 wt %) was prepared by incipient wetness impregnation method. Platinum (II) acetylacetonate was used as precursor. In order to locate platinum particles mainly at the exterior surface of SiO₂; the pores of SiO₂ support was prior saturated with n-hexane. After that, a certain amount of platinum (II) acetylacetonate solution was added to SiO₂ powder drop by drop. The prepared catalyst sample, after controlled impregnation in a sonication bath for 1 h, was dried in vacuum at 323 K. The sample was finally calcined at 673 K for 4 h in a programmable furnace with a heating rate of 290 K/min.

The prepared catalyst was characterized by nitrogen adsorption, SEM, IR and XRD measurements. SEM image of Pt/SiO₂ catalyst showed that the distribution of Pt particles on the support was uniform and not agglomerated. The nitrogen adsorption isotherm of catalyst is of type IV according to IUPAC classification. BET surface area, Langmuir surface area, macro + meso pore surface area and average pore diameter of the catalyst were measured as 297.72 m²/g, 419.16 m²/g, 273.88 m²/g and 7.9 nm, respectively. In the XRD patterns of Pt/SiO₂ catalyst, it was observed that SiO₂ exhibited a halo around at 2θ of 22°, the peaks at 2θ = 25.4°, 29.0°, 31.4° and 38.8° were attributed to gypsum which was an impurity (5%) in silicagel. The peaks characterizing Pt crystals were

observed at 2θ of 39.8°, 46.3° and 67.2°. IR study of the catalyst before and after the reaction showed that structure of the catalyst did not change during the reaction.

In the oxidation of AA over Pt/SiO₂ catalyst, the effects of initial concentration of AA, catalyst loading and temperature were studied. The samples taken periodically from the reaction mixture were analyzed by a gas chromatograph with a DB-WAXetr capillary column. Degradation degree of AA increased from 25.0 to 48.4 % with decreasing initial concentration from 3000 ppm to 1000 ppm. To investigate the effect of catalyst loading in CWAO of acetic acid, experiments were carried out at 333 K in the presence of air with a flow rate of 3 ml/s for initial concentration of 3000 ppm with different catalyst amounts such as 0.5, 1.0 and 1.5 g. The initial specific rates measured as a function of the mass of catalyst were almost similar at about 1×10^{-5} mol/Ls for catalyst loadings of 1 and 1.5 g/150 mL. It showed external mass transfer resistance was negligible in that catalyst loading range. Internal diffusion resistance was also negligible due to the catalyst used in powder form. The effect of temperature on CWAO of acetic acid was tested at three different temperatures, namely at 328, 333 and 338 K. The results showed that initial rate of reaction increased with temperature in the range of 328 to 338 K. Due to the decrease in solubility in O₂ with temperature, after a reaction time of 3 h, conversion of AA dropped significantly for the reaction temperature of 338 K. It means that, to avoid mass transfer limitation of O₂, temperatures lower than 338 K should be used in AA oxidation under the conditions in this study. CWAO reaction of AA was described by an empirical kinetic model with an activation energy of 141.77 kJ/mol.

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Adsorption Efficiency of Chitosan Nanofibers Towards Heavy Metal Ions at Low Concentration

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Abstract- Electrospun chitosan nanofibers were used as adsorbent for removal of cadmium(II), copper(II), iron(III) and silver(I) ions from aqueous solutions. The fiber was characterized by electron microscopy and the sorption of metals by chitosan nanofibers was examined by using inductively coupled plasma mass spectrometry (ICP-MS). Sorption percentage as a function of sorbent amount, shaking time, and initial metal ion concentration was investigated by both batch equilibration and membrane filtration processes.

Nonwoven nanofiber technology attracts increasing attention due to the small size of the fibers that provides much enhanced and even excellent functions which can not be obtained by bulk materials. Among the techniques for fabrication of nonwoven fabrics such as drawing, template synthesis, phase separation, self-assembly, etc. electrospinning is one of the more commonly used methods for the continuous production of nanofibers with considerable characteristics such as controllable fine diameters on the order of a few tens to hundreds of nanometers, i.e. high surface area to mass ratio, mechanical integrity, and ease of fabrication. These properties provide a number of potential applications such as filtration [1,2], biosensors [3], drug delivery [4], wound dressing [5], and protective clothing [6].

Ground water pollution associated with both natural and anthropogenic processes is a serious problem in many countries. Manmade reasons such as mining, plating, glass making, ceramics, and battery manufacturing cause a great increase in heavy metal contamination in water. In addition, the metal ions are released into environment from a variety of natural sources including mineral and metallic deposits in sedimentary rocks and soil. The elevation of heavy metal concentrations in ground water reveals a significant risk to public health and environment.

The adsorption/separation of trace metal ions from aqueous solutions has appreciable significance in order to control the contamination problems by removal of toxic metals. The removal of metals can be managed by using adsorbents including functional groups that have metal binding ability such as amino, carboxyl, phosphoric, mercapto, and amidoxime, etc. Chitosan is a suitable natural biopolymer for filtration application, thus the amino groups and hydroxyl groups are covalently attached onto the chitosan backbone [7].

In this study, chitosan nanofibers with average fiber diameter of ~40 nm were prepared via an electrospinning technique. BET surface area of the medium molecular weight chitosan powder was increased from 0.92 m²/g to 22.4 m²/g by transforming into nanofibers. This indicates a higher surface area, therefore higher amounts of available free amine groups, which are expected to offer a higher sorption capacity.

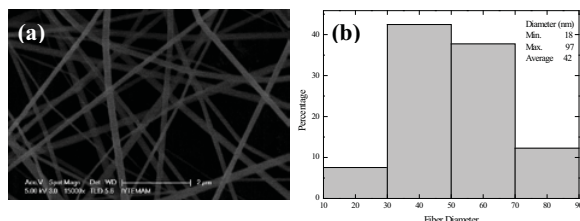


Figure 1. (a) SEM micrograph of chitosan nanofibers electrospun from a 0.4 wt% solution and (b) diameter histogram of nanofibers

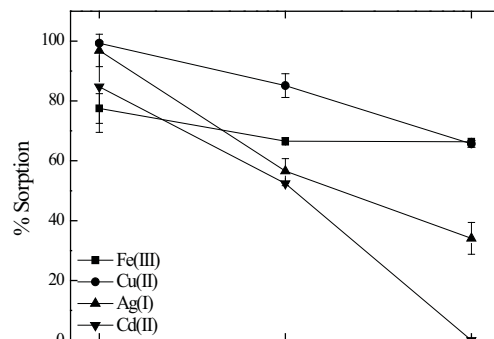


Figure 2. Effect of initial concentration on the adsorption of Fe(III), Cu(II), Ag(I), Cd(II) on the chitosan nanofibers

Figure 1 presents a uniform nanofibrous mat obtained from 0.4 % (wt/wt) chitosan solution in HFIP by electrospinning in the electric field of 1.1 kV/cm.

Figure 2 shows the adsorption percentage of Fe(III), Cu(II), Ag(I), Cd(II) ions by chitosan nanofiber mat as a function of initial concentration.

The order of adsorption efficiency of metal ions was found to be in the order of Cu(II) > Fe(III) > Ag(I) > Cd(II). The preliminary results show that the chitosan nanofibers have higher metal binding capacity compared to chitosan powder. The filtration yield is related to the materials used for filter. Therefore, their high surface area, small pore size, and mechanical integrity make electrospun textiles as good alternatives in membrane filtration processes.

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Improvement of the Electrochemical Hydrogen Storage Performance of Mg-Based Alloys Synthesized by Mechanical Alloying

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Abstract— Al, Zr and Ti including Mg-Based alloys were synthesized by mechanical alloying and their electrochemical hydrogen storage characteristics were investigated. X-ray diffraction studies showed that while Al was retarding, Zr and Ti were facilitating the amorphization of the main phase. Although Al-including alloy had very low initial discharge capacity, the capacity retaining rate of this alloy was much better than those of Ti- and Zr-including alloys. The presence of Ti-oxide were predicted to make Mg(OH)₂ barrier layer more penetrable by hydrogen atoms, since despite the increased stability of the surface layer the cyclic stability of Ti-including alloy was relatively satisfactory.

Magnesium-based hydrides are very promising hydrogen storage materials due to their high theoretical capacity, low cost and light weight, and they are known as environmental friendly. The poor hydriding/dehydriding kinetics at room temperature, however, hampers the practical applications of these alloys. The intensive efforts have been spent to improve the kinetic properties of the Mg-based alloys. The most significant improvement was realized with the synthesis of these alloys by the mechanical alloying [1]. The further improvement in the hydriding/dehydriding performance of the Mg-based alloys, which are synthesized by the mechanical alloying, can be achieved by the partial replacement of Mg and/or Ni in the main phase. Among the replacement elements, aluminum [2-4], zirconium [2-5] and titanium [4-5] have attracted considerable attention. In this work Mg₂Ni, Mg_{1.5}Al_{0.5}Ni, Mg_{1.5}Zr_{0.5}Ni and Mg_{1.5}Ti_{0.5}Ni alloys were synthesized by mechanical alloying at the same conditions and their electrochemical hydrogen storage properties were compared.

The discharge capacities of Mg₂Ni, Mg_{1.5}Al_{0.5}Ni, Mg_{1.5}Zr_{0.5}Ni and Mg_{1.5}Ti_{0.5}Ni alloys are 258, 166, 322 and 414 mA h g⁻¹, respectively, in Figure. Mg₂Ni alloy can able to keep only about 10% of its initial discharge capacity at 20th cycle. Mg_{1.5}Ti_{0.5}Ni alloy has the highest discharge capacity value at all the cycle numbers and it is able to keep about 34% of its initial discharge. Although the cycling performance of Mg_{1.5}Zr_{0.5}Ni alloy is much better than that of Mg₂Ni alloy, both the initial discharge capacity and capacity retaining rate of this alloy are not as good as those of Mg_{1.5}Ti_{0.5}Ni alloy. Despite the presence of very low initial discharge capacity, Mg_{1.5}Al_{0.5}Ni alloy keeps almost 45% of its initial capacity at 20th cycle. Apparently the high Al content prevents the rapid degradation of Mg₂Ni phase with the charge/discharge cycles.

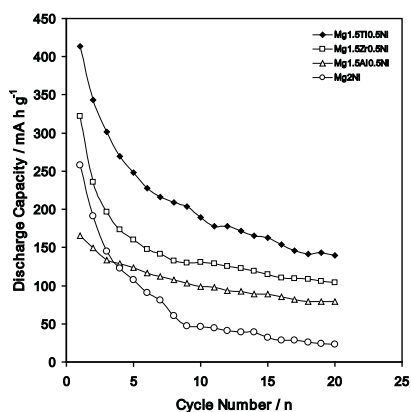


Figure. Discharge capacities as a function of cycle number.

It is agreed that the rapid degradation in the discharge capacity during the charge/discharge cycling of Mg₂Ni phase results from the formation and thickening of Mg(OH)₂ surface layer, which retards the hydrogen in and out diffusion, in alkaline solutions [1-5]. Probably the presence of Al in the alloy decreases the stability of Mg(OH)₂ surface layer and helps the hydrogen diffusion. In fact it was proved by the studies include the XPS characterizations of the Al-containing Mg-based hydrogen storage alloys that Al₂O₃ layer, which is highly unstable in the strongly alkaline solutions, can able to form on the alloy surface [6]. The selective dissolution of the disseminated Al₂O₃ can make Mg(OH)₂ surface layer porous and more permeable for hydrogen. Also the formation of the porous layer may expose the underlying electrocatalytically active Ni sites and helps the hydriding/dehydriding electrochemical kinetics [2-4].

The discharge capacities of the Ti- and Zr-including alloys are very promising due to their improved charge/discharge reversibility. Since Ti is a hydride forming element like Mg, the alloy discharge capacity is much higher in the presence of Ti. The previously predicted action of Ti during the alloy cycling was that Ti segregates to the surface and makes the alloy surface more penetrable by atomic hydrogen [5]. There is no direct experimental evidence to support this proposal. Our observations, however, can be explained with this proposal that Ti improves the alloy cyclic stability. The oxide of Zr may not be very stable in alkaline solutions like Al-oxide and the selective dissolution of the disseminated Zr-oxides may again reduce the stability of the barrier Mg(OH)₂ and thus contributes positively to the alloy cyclic stability.

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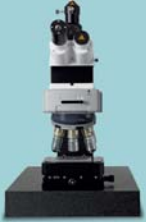
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A New Nanoporous Material Family for Membrane-based Gas Separations: Metal Organic Frameworks

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Abstract- A nanoporous metal organic framework (MOF) material is identified with a very high CO₂/CH₄ selectivity. Molecular simulations and continuum modeling have been used to predict the performance of both pure MOF membranes and polymer/MOF composite membranes for separation of CO₂ from CH₄.

Membranes offer a promising approach for energy-efficient separation of gas mixtures. One challenge in membrane-based separation applications is to determine the membrane material among a wide spectrum of available materials. Large numbers of nanoporous materials exist that are potentially suitable for use as gas separation membranes.

Metal organic frameworks (MOFs) are a relatively new class of nanoporous materials which exhibit several advantages over traditional nanoporous materials for gas adsorption and gas separations. MOFs can be synthesized with exquisite control over pore shape and connectivity [1]. Thousands of distinct MOF materials have now been synthesized. However, very little is known about the performance of these nanoporous materials as membranes in gas separations [2].

In this study, atomistically detailed simulations have been used to predict the performance of MOF membranes for separation of carbon dioxide from methane, which is an important issue in natural gas purification. Identification of membrane materials with high selectivity for carbon dioxide/methane mixtures could revolutionize this industrially important separation.

Computational methods including grand canonical Monte Carlo simulations, molecular dynamics, transition state theory showed that carbon dioxide diffuses ~2500 times faster than methane in a MOF, Cu(hfipbb)(H₂hfipbb)_{0.5}, since the kinetic diameter of methane is very similar to the pore size of this MOF.[3] Molecular simulation results yielded that a membrane based on this MOF would have a net selectivity for CO₂ of 10⁴-10⁵, which is significantly higher than any known polymeric membrane or high performance zeolites membranes. [3]

One advantage of MOFs is that these highly selective nanoporous structures can be used as additives in polymer membranes to increase the selectivity of the polymer membranes. Therefore, composite membranes in which nanoporous MOF, Cu(hfipbb)(H₂hfipbb)_{0.5} particles are embedded into a polymer, Matrimid has been also computationally studied using Maxwell and Bruggeman models. Figure 1 shows that adding Cu(hfipbb)(H₂hfipbb)_{0.5} into Matrimid enhances both the selectivity and the permeability of the Matrimid membrane. For example, 30% loading of this MOF in Matrimid increases the permeability (selectivity) of CO₂ from 9 (40) to 20 (100) Barrer [4].

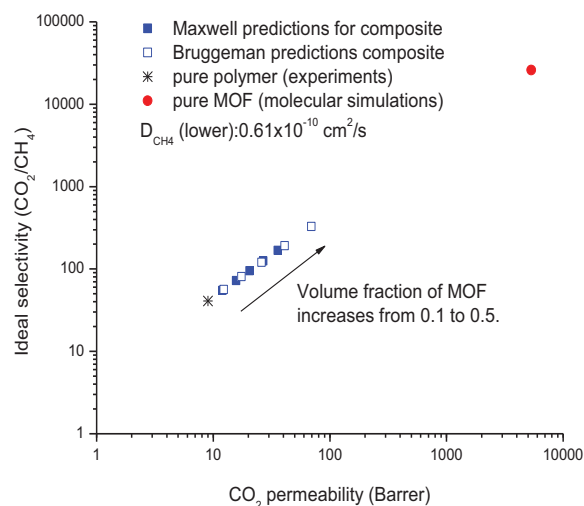


Figure: Ideal selectivity for CO₂ as a function of gas permeability for pure polymer membrane, pure MOF membrane and composite membranes made of polymer and MOF [4].

This study concluded that MOFs can be used for kinetic separation of gases based on the pore sizes of the material and using a highly selective MOF such as Cu(hfipbb)(H₂hfipbb)_{0.5} in composite membranes can dramatically improve the membrane's performance relative to pure polymer.

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