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Cyclodextrin-Modified Iron Oxide

Iron oxide is common in the nature. There are several forms of minerals containing ferrous and ferric oxides often mixed with hydroxides. More than 90% mined is used for steel production. The application as pigment is also important.

Recently new applications emerged: iron oxide in the form of nanoparticles (1-100 nm) has superparamagnetic properties and useful for terabit magnetic storage devices, catalysis, sensors, and high-sensitivity biomolecular magnetic resonance imaging (MRI) for medical diagnosis and drug delivery. These nanoparticles are effective sorbents useful also in water purification. The main advantage of these nano-sized magnets is the simplicity of the usage and efficiency (they can be targeted, collected by external magnetic field). In this literature review, the potential of cyclodextrins for improving the characteristics of iron oxide has been overviewed.

Catalyst

Oxidation of 2,4,6-trinitrotoluene (TNT) by hydrogen peroxide is catalysed by iron-containing minerals (Fenton reaction). The catalytic effect is higher with the minerals of higher iron content. Magnetite, a mineral (ferrous-ferric oxide) of over 70% iron (Fe II and Fe III) content was found effective catalyst[1]. The efficacy was further enhanced in the presence of carboxymethyl β-cyclodextrin (CMBCD), which enhanced the dissolved iron concentration. Compared to other iron-chelating agents, such as EDTA the degree of TNT mineralization was twice higher in the presence of CMBCD than in the presence of EDTA. This enhancement could in part be due to the increase in dissolved iron concentrations in the bulk solution in addition to the enhancement of the solubility (availability) of the poorly soluble contaminants. Similar results were obtained for polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs) using CMBCD[2] as well as for TNT/methyl BCD[3], pentachlorophenol (PCP)/CMBCD[4], trichloroethylene (TCE)/HPBCD[5,6] TCE/RAMEB[7], benzene/HPBCD[8] systems.

BCD conjugated Fe_3O_4 magnetic nanoparticles (Figure 1A) were an efficient microvessel system for nucleophilic substitution reaction of benzyl halides in water. No evidence for the formation of by-product, such as isothiocyanate or benzyl alcohol was observed and the products were obtained in pure form without further purification. The nanomagnetic catalyst could be readily separated from solution via application of an external magnet, allowing straightforward recovery and reuse[10].

Silica coated with iron oxide and modified by CD or its derivatives or complexes is an efficient catalyst to oxidize carbon monoxide (CO) to carbon dioxide[11]. The catalyst is useful in the apparatus used for the removal of CO from fuel cell air intake and from enclosed space.

A magnetically separable catalyst was synthesized via a carbodiimide activation process to get CD-functionalized core-shell nanoparticles (Fe_3O_4/SiO_2/CMBCD) (Figure 1B). The catalyst presented high activity for selective oxidation of various alcohols with NaOCl as oxidant and water only as the solvent[9].

Iron oxide/titanium oxide core-shell nanoparticles modified by CDs are efficient photocatalyst for wastewater treatment. The amorphous TiO_2 shell accelerates the degradation and mineralization of the organics, such as bisphenol A and dibutyl phthalate, under UV illumination, and the magnetism associated with the crystalline Fe_3O_4 core allows the magnetic separation from the dispersion once photocatalytic degradation is complete. The tethered CDs are responsible for the aqueous dispersibility of the nanoparticles and their hydrophobic cavities for the capture of the organic pollutants that may be present in water samples[12].

High conversion and enantioselectivity were achieved by *Candida rugosa* lipase encapsulated by silica-functionalized BCD grafted on the surface of iron oxide nanoparticles[13]. The hydrolysis of racemic Naproxen methyl ester resulted in 98% S- Naproxen.

Sensors and analytical applications

An amperometric sensor based on coating glassy carbon electrode by iron oxide in the presence of BCD was developed for the analysis of bisphenol A[14]. Enhanced (two orders of magnitude) linearity, improved (sub-micromolar) detection limit, and reproducibility make this sensor potentially exploitable for analysis of real samples, such as food and beverages.

Mono-6-formyl-BCD moieties were attached to (3-aminopropyl)triethoxysilane-coated superparamagnetic Fe_3O_4 nanoparticles (Figure 1B) and the oligosaccharide-capped core-shell nanoparticles were employed as support for the supramolecular immobilization of two different adamantane-modified enzymes, tyrosinase and xanthine oxidase, through host-guest interactions. The enzyme-modified nanomaterial was further used to magnetically modify carbon paste electrodes for constructing amperometric biosensors toward cathecol and xanthine[15].

Iron oxide-silica core-shell particles modified with CMBCD can be used for sample pretreatment and analysis of samples containing nucleosides and amino acids, respectively. The immobilized CMBCD gives the potential for selective binding of guanosin compared to adenosine and enantiomers of amino acids (Tyr, Trp, Phe)[16,17].

Drug delivery and magnetic resonance imaging (MRI)

BCD-functionalized magnetic Fe_3O_4 nanoparticles have been prepared by a two-step anchoring route based on particle prefunctionalization with a phosphonic monolayer, which acts as a covalent linker between the nanoparticles and BCD (Figure 1A). The ability of nanoparticles to carry and slowly release some drugs was tested by using diclofenac sodium salt as a model[18].

Silica-iron oxide hybrid nanoparticles were modified either with fluorescent moieties for imaging, or various functionalities for targeting or by CDs to improve the drug binding and stability. For instance, CD-modified nanoparticles (Fe-Si-CD-PEG) are an excellent platform for anticancer drug delivery and MR imaging[19]. The glutathione (GSH)-responsive CD gatekeepers on the surface of the hybrid nanoparticle play a key role in accommodating anticancer drug molecules in the pore of the silica shell without premature release until CD gatekeepers are cleaved by GSH. It was confirmed, from an in vitro study with the A549 cell line, that doxorubicin (DOX) was released from the internalized carriers due to GSH-mediated cleavage of the CD gatekeeper resulting in apoptotic and clonogenic cell death. The accumulation of Fe-Si-DOX-CD-PEG in the tumors was detected by *in vivo* MR imaging. The growth of the tumor *in vivo* was effectively suppressed by the intravenously injected Fe-Si-DOX-CD-PEG.

Badruddoza *et al.* synthesized silica-iron oxide hybrid core-shell magnetic nanoparticles with quadrupole functionalities by anchoring fluorescent and targeting moieties together with CD (fluoresceinyl isothiocyanate, FITC, folic acid, FA and CMBCD, Figure 1C)[20]. This smart theranostic system was useful for simultaneous fluorescence imaging, magnetic manipulation, cancer cell-targeting and hydrophobic drug (retinoic acid) delivery.

A novel approach to thermo- and magnetoresponsive drug delivery was presented by Marten *et al.*, using magnetic nanoparticles decorated with a BCD-containing polymer brush shell (Figure 2B). The iron oxide based magnetic cores with a diameter of 10-12 nm can be used for remote controlled magnetic heating. The polymer shell is created by atom transfer radical copolymerization of various methacrylates and the methacrylate functionalized BCD monomer. The BCD units are responsible for a thermosensitive complex formation with drugs[21]. Another variety of the polymer shell is gum arabic fixed on the iron oxide surface[22]. BCD is grafted to this polymer shell with a diisocyanate. The controlled release of retinoic acid from these nanoparticles was observed.

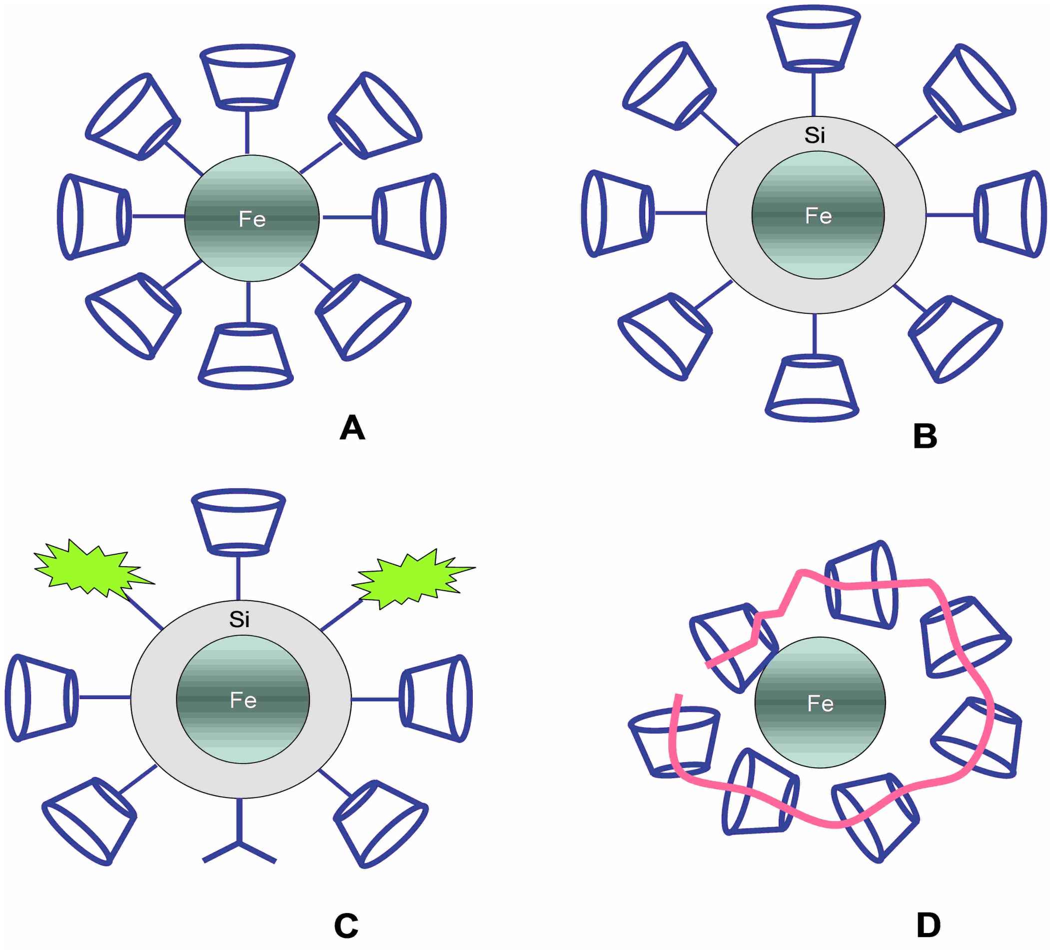

Figure 1 Schemes of the various CD-modified iron oxide nanoparticles: CDs attached to the iron oxide core (A), to the iron oxide-silica core-shell (B), iron oxide-silica core shell nanoparticles with multiple functionalities (fluorescent, targeting and CD moieties) (C), iron oxide coated with polyrotaxane (D)

Superparamagnetic iron oxide nanoaggregates (SPIONs) were surface-coated with amine functionalized polyrotaxane and were proposed as a carrier for inhalation dry powders (Figure 1D). Polyrotaxane is primarily composed of BCD threaded on the block copolymer, poly(propylene glycol) bis(2-aminopropylether)[23] or polyethylene oxide-polypropylene oxide copolymer (Pluronic)[24]. These aggregates in combination with a target-directed magnetic gradient field showed promise for targeted pulmonary deposition of anticancer drugs. If loaded with a pharmaceutical active ingredient, these particles may be useful for treating localised lung disease such as cancer nodules or bacterial infectious foci[25].

Starlike polymers with multiple dextran arms were designed and developed by attaching dextran to a BCD core through click chemistry. Next, starlike dextran was modified with aliphatic chains and these amphiphilic polymers can self-assemble into nanoscale micelles, which can encapsulate multiple superparamagnetic iron oxide nanoparticles (Figure 2A). The resulting nanocomposites have a high relaxivity under a clinical MRI scanner. Further, dual functional probes were developed by loading both superparamagnetic iron oxide nanoparticles and small molecule anticancer drug doxorubicin into polymeric micelles. Multidrug-resistant breast cancer cells MCF-7/Adr treated with these probes can be characterized under MRI[26].

Sorbent

The main advantage of using iron oxide as sorbent in waste water purification is that magnetic nanoparticles can be easily separated from the waste water by applying magnetic field. The pollutants sorbed on the surface can be removed, and the recovered nanoparticles can be used in several cycles. Modifying the surface of the magnetic nanoparticles with CDs result in enhanced sorption of organic/inorganic pollutants.

Sorbent for removal of toxic substances from blood during hemoperfusion is one of the potential applications of such magnetic nanoparticles modified with CDs. Diazepam, as model toxin was removed from blood using BCD-conjugated magnetic nanoparticles[27].

Grafting CD to the surface of magnetite nanoparticles via functionalization with [3-(2,3-epoxypropoxy)propyl]trimethoxysilane moieties resulted a sorbent able to remove various dyes, such as Direct Blue 15, Evans Blue, and Chicago Sky Blue from effluents[28].

CMBCD was grafted on the surface of magnetic nanoparticles covered by silica shell via carbodiimide method[29,30]. The resulting modified nanoparticles showed enhanced sorption of methylene blue and metals (Cu, Pd, Ni and Cd) from aqueous solutions. The maximal sorption capacity reached 278, 47 65, 28 and 13 mg/g, respectively. Enhanced CD concentration can be achieved on the surface of the particles by using BCD polymer prepared by cross-linking CMBCD with epichlorohydrin (Figure 2B)[31].

BCD modified multiwall carbon nanotubes/iron oxide is a promising magnetic material for the preconcentration and separation of toxic metals, such as Cu and Pb as well as organic pollutants (1-naphtol) from aqueous solutions in environmental pollution cleanup[32,33].

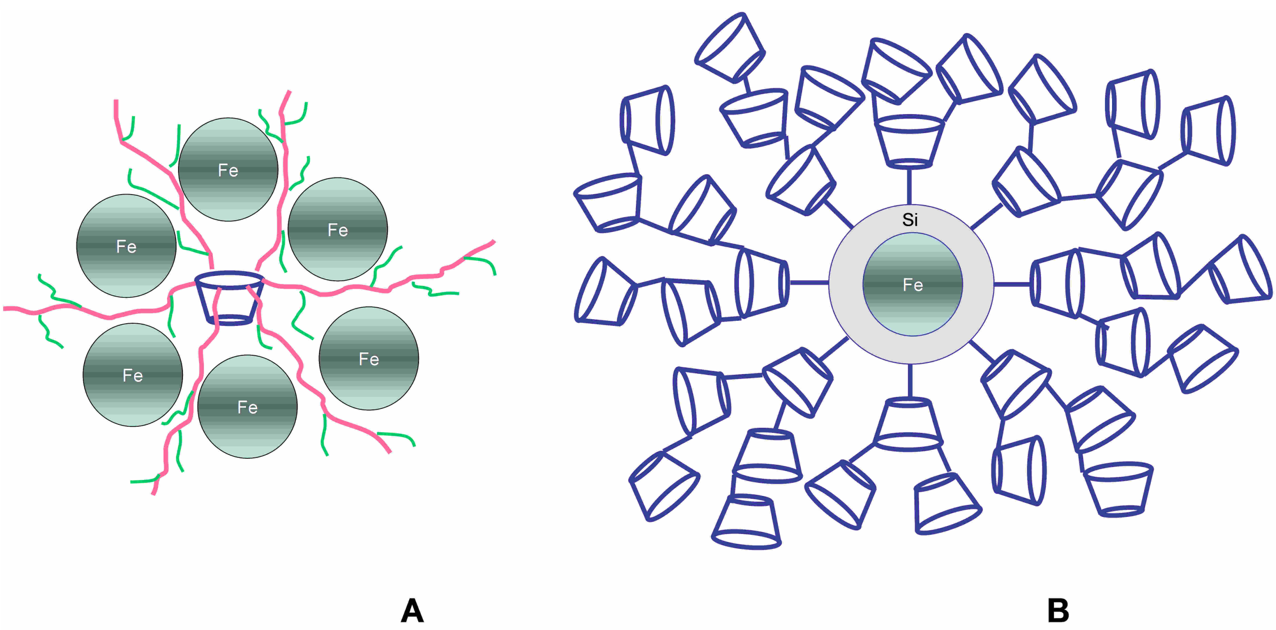

Figure 2. Scheme of iron nanoparticles embedded in starlike dextran polymer substituted with alkyl groups (A) and iron oxide-silica core-shell nanoparticles grafted with CD polymer (B)

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Eva Fenyvesi

CycloLab Cyclodextrin R&D Laboratory, Ltd.,

Budapest, HUNGARY

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Binding Constants, Diacerhein, Hydrolytic Degradation, Molecular Spectroscopy

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Cellular delivery, Chitosan, Curcumin, Spray drying

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Dothiepin, Doxepin, Inclusion Complex, Supramolecule, Nano structure

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Cyclodextrins, Inclusion complex, Nano sheet, Nanomaterial, Self assembly, Sulfa drugs

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Bupivacaine, Inferior Alveolar Nerve Block, Local Anaesthesia, HPBCD

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In vitro Release Studies, Membrane Transport, Tetracaine, (2-Hydroxy)propyl-β-CD, Super-Case Transport Mechanism

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Carvedilol, Dissolution Enhancement., Ionization Process, Tartaric Acid, Water Soluble Polymer, HPBCD, PVP, Poloxamer

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Cytochrome C, Graphene, Graphene–Cyclodextrin–Cytochrome C, Supramolecular Recognition, Graphene Nanosheets, Mimic the Confined Environments of the Intermembrane Space of Mitochondria

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Biotransformation, Cyclodextrin, DHEA, Solubility, Improvement of Cell Permeability

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Active Ingrediants., Cosmetic Textiles, Denim Fabric, Sodium Carbonate, β-Cyclodextrin

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Adsorption, Emerging Contaminant, Hybrid Adsorbent, Water Treatment, β-Cyclodextrin, 17β-Estradiol, Perfluorooctanoic Acid, Bisphenol-A, Steroid Hormones

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Click Reaction, Cyclodextrin, Electrospinning, Filtration, Nanofibers, Phenanthrene

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Industrial Effluent, Oxidation, Pollutant, Sorption, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD)

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Nanostructure, Recombination, Structural Materials, Dye-Sensitized Solar Cells, 4-Methyl-1-Cyclohexane Carboxylic Acid

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Ballpoint Pen Ink, Inclusion Complex, Sun Protection Factor, UV Absorber, β-Cyclodextrin, UV Protection, Photo Degradation

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Catalysis, Chemzymes, Hydrogen Peroxide, Michaelis–Menten, Peroxidase

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> *Characteristic Temperature, Characteristic Thermal Constant, Cyclodextrin Stationary Phase, Enantioselective Gas Chromatography, Optimal Heating Rate*

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Alkaloid, Berberine, Capillary Electrophoresis, HPBCD, Plant, Tablets

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DIMEB, Baicalin, Electrochemical Sensor, Graphene, Isoquercitrin, Monitoring of Ultra-Trace Flavonoid Drugs

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Chrysoidine, Electrochemical Sensor, Electropolymerization, Molecular Imprinting

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Ligation-Rolling Circle Amplification, Single-Nucleotide Polymorphism, Stemless Molecular Beacon, γ-Cyclodextrin, Bis-Pyrene Labeled DNA, Recognition of a Point Mutation DNA Targets

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CYCLOLAB

Edited and produced by: CYCLOLAB
Homepage:www.cyclolab.hu
H-1525 P.O. 435, Budapest, Hungary
Tel:(361) 347-6060
Fax: (361) 347-6068
e-mail: cyclolab@cyclolab.hu