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Electrospinning of Functional Nanofibers with Cyclodextrins

Functional nanofibers by electrospinning

Electrospinning is a versatile and cost-effective technique for producing functional nanofibers from various polymers, blends, composites, ceramics, etc. Electrospinning is an electro-hydrodynamic process which has lately become an exciting and versatile platform technology for the production of nanofibrous materials. The simplicity of the electrospinning setup and the relatively high production rate of nanofibers make this process highly attractive for both academia and industry. In electrospinning technique, a continuous filament is electrospun from polymer solutions (most common) or polymer melts (very limited) under a very high electrical field (Figure 1a) resulting in ultra-fine fibers (1000 times smaller than a single human hair) ranging from tens of nanometers to few microns in diameter (Figure 1b). Very briefly, the electrospinning process takes place between a spinning head (capillary opening (nozzle) or a rotating drum (nozzle-less)) and a collector counter electrode where a high voltage is applied (typical 10-30 kV) (Figure 1a). Electrospun nanofibers/nanowebs (Figure 1c) have several remarkable characteristics such as very large surface-to-volume ratio, high porosity within the nanoscale, unique physical and mechanical properties along with the flexibility for chemical/physical functionalization. The outstanding properties and multi-functionality of nanofibers/nanowebs make them favorable candidates in many areas including healthcare, filtration, textiles, energy, sensors, electronics, environmental, food, packaging, agriculture, etc.

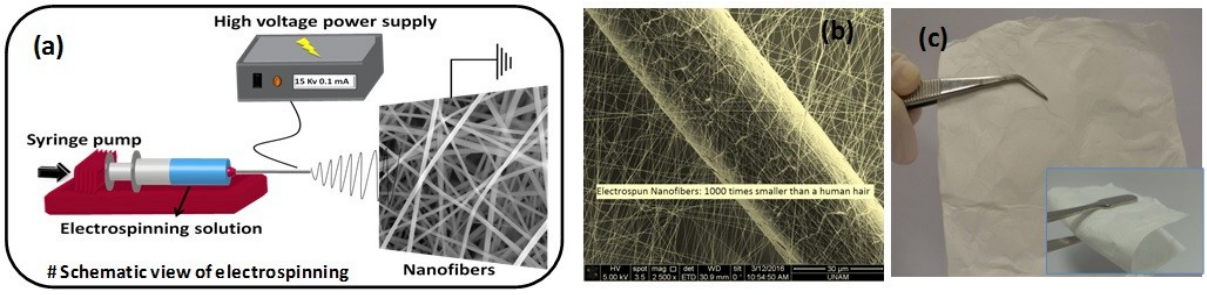

Figure 1. (a) Schematic view of electrospinning,
(b) Electrospun polymeric nanofibers (~50-100 nm diameter) on my single hair (50 micron),
(c) Free-standing and flexible nature of electrospun nanofibrous mat.

Functionalization of electrospun nanofibers with cyclodextrins

The functionalization of nanofibers with cyclodextrins (CDs) and cyclodextrin inclusion complexes (CD-ICs) is extremely attractive since electrospun nanofibers/nanowebs containing CDs and/or CD-ICs will have unique characteristics that can potentially improve and broaden the application areas of cyclodextrins and electrospun nanofibers/nanowebs. In our research, the electrospinning of functional nanofibers with CDs and/or CD-ICs is of particular interest to us, since functional nanofibers/nanowebs can be produced for various application areas such as filtration, medical, food and food packaging, textile, agriculture, etc.

Cyclodextrin functionalized electrospun polymeric nanofibers

By means of the electrospinning technique, we have successfully produced CD functionalized polymeric nanofibers (Figure 2a). Various polymers such as polystyrene (PS) [1], polymethylmethacrylate (PMMA) [2], polyethylene oxide (PEO) [3] and zein [4] were blended with CDs and electrospun into uniform nanofibers/nanowebs. Three different types of native CDs; α-CD, β-CD and γ-CD were incorporated individually in electrospun polymeric nanofibers. We found that the addition of CD in the polymer solutions facilitated the ability to electrospinning nanofibers from polymer solutions at low polymer concentration yielding in bead-free and uniform nanofibers. We observed that CD molecules were homogeneously distributed within the polymer matrix without forming phase separated crystalline aggregates. The choice of polymeric matrix is important in which polymer chains should not make inclusion complexation with CD cavity and therefore CD will be available for further complexation depending on the application such as encapsulating organic pollutants for filtration.

Molecular filters based on cyclodextrin functionalized electrospun polymeric nanofibers

We have reported a new method for taking advantage of the very high surface area of electrospun nanofibers to form molecular filters and/or nanofilters, with the added advantage of incorporating CDs as functional additives that can trap molecules and function as highly efficient filtration systems. The electrospun nanofibrous mats can both physically filter tiny particles and remove industrial waste. The combination of CDs and the electrospun nanofibers can potentially increase the efficiency of filters by facilitating complex formation with organic compounds and the very high surface area of the nanofibers. We have demonstrated that CD molecules can be incorporated onto electrospun nanofibers/nanowebs in order to develop more efficient filters by facilitating both inclusion complex formation of CD with hazardous organic molecules and the very high surface areas of the electrospun nanowebs. The molecular filtration capability of these CD functionalized nanofibers/nanowebs was utilized for the removal of organic waste molecules from aqueous solutions. As a proof-of-concept study, CD molecules were successfully incorporated in electrospun polystyrene (PS) nanofibers and these

CD functionalized nanofibrous membranes showed potential for efficient removal of organic compounds (e.g.: phenolphthalein) from solution by the formation of inclusion complexes with the CD molecules [5-6]. But, most of the CD molecules were buried inside the fiber matrix and cannot be efficiently used for entrapment of pollutant molecules, only some CD molecules were present on the fiber surface. Moreover, CD molecules were physically attached to the fiber surface; so, the leaching of CD molecules from the fiber surface during filtration especially in the liquid media was problem [5-6]. To overcome the leaching of CD in water filtration, the surface modification of nanofibers with crosslinked poly-cylodextrin [7] or the chemical attachment of CD onto electrospun nanofibers by click reaction [8] was successful, and the removal of phenanthrene (PAH molecule) from aqueous solution was achieved [7-8]. We have also shown that the electrospun nanofibers from physical mixture of polymer/CD can be quite applicable for air filtration, for example, removal of volatile organic compounds (VOC) such as aniline, styrene and toluene, from the surroundings (Figure 2b) [9-11]. Our results demonstrate that nanofibers produced by electrospinning that incorporate CDs (α-CD, β-CD and γ-CD) with different sized cavities can filter organic molecules and the efficiency of the filtration was dependent on the type of the CD used [6-11], and therefore, these CD functionalized electrospun nanofibrous membranes can potentially be used for filtration, purification, and separation processes.

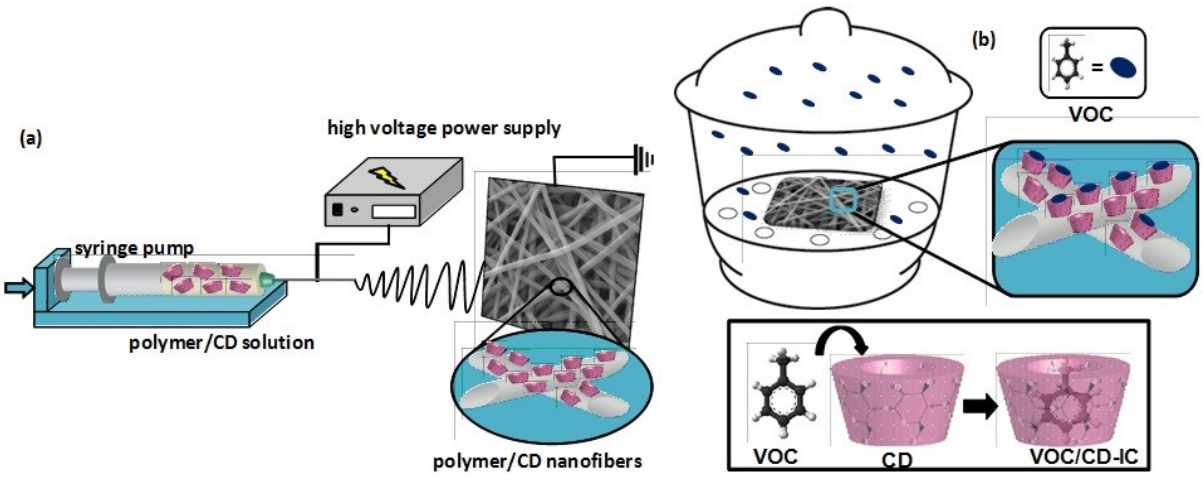

Figure 2. (a) Schematic representation of the electrospinning of polymer/CD nanofibers, (b) Schematic representation of VOC removal by electrospun polymer/CD nanofibrous web for air filtration application [9-11].

Cyclodextrin functionalized electrospun polymeric nanofibers/nanowebs: High temperature stability and slow release of volatile active agents and controlled release of drugs

Bioactive and volatile additives such as fragrances/flavors, essential oils, antioxidants, drugs, antibacterials are mostly sensitive to heat, oxygen and light, however, cyclodextrin inclusion

complexation (CD-IC) is very effective for the stabilization/protection and controlled/sustained release of these additives. Therefore, incorporation of CD-IC into electrospun nanofibrous matrix would improve the shelf-life, stability and slow release of such active additives. Controlled release of drugs and antibacterials can also be achieved effectively by CD-IC. Our research group has been very active in formation of CD-IC with certain additives such as fragrances/flavors (menthol [12-14], vanillin [15-16]) antibacterial agents (triclosan [17], allyl isothiocyanate [18]) essential oils and antioxidants (eugenol [19], geraniol [20], quercetin [21], gallic acid [22], α-tocopherol (Vitamin-E) [23-24], curcumin [25]) and drugs (naproxen [26], sulfisoxazole [27]) (Figure 3). Then, these CD-ICs were incorporated in polymeric electrospun functional nanofibers/nanowebs. With this approach, functional nanofibrous materials for textile, medical textile, food and packaging applications can be obtained having long-lasting functionality due to stabilization and sustained/controlled release of these additives by CD complexation.

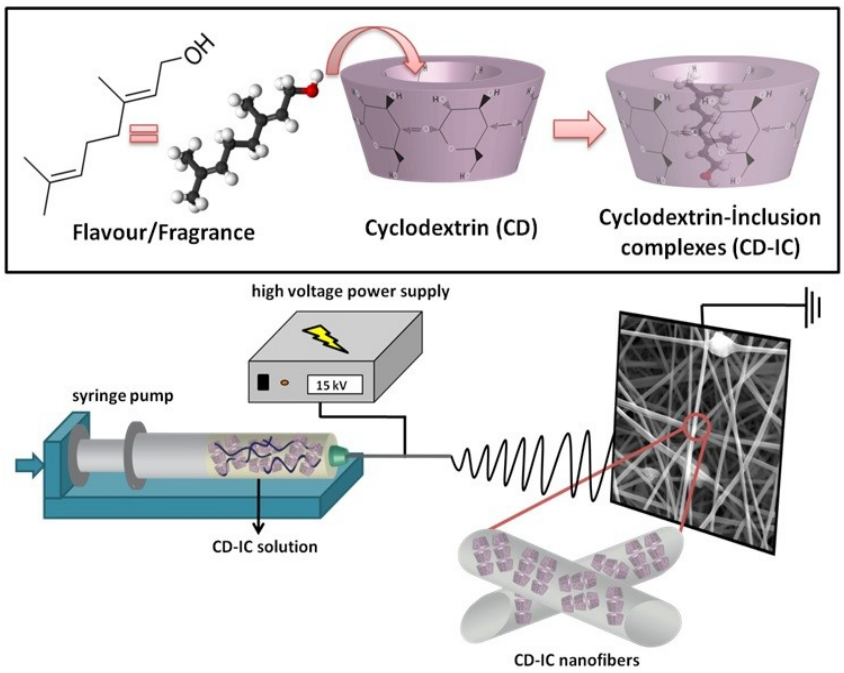

Figure 3. Schematic representation of inclusion complex formation between CD and guest molecule (active agent such as flavor/fragrance) and the electrospinning of polymer/CD-IC nanofibers.

Electrospinning of nanofibers from cyclodextrins

In principle, electrospinning of nanofibers involves high molecular weight polymers and high solution concentrations since entanglements and overlapping between the polymer chains sustain the continuous stretching of electrified jet for uniform fiber formation, otherwise, for small molecules, electrospraying occurs which yields only beads instead of fibers. Hence, the electrospinning of nanofibers from non-polymeric systems is quite a challenge. Cyclodextrins

are cyclic oligosaccharides which are capable of self-assembly and form aggregates via intermolecular hydrogen bonding in their concentrated solutions. In our earlier study, we performed electrospinning of cyclodextrin-pseudopolyrotaxane (α-CD/PEG) nanofibers but we needed to use polymeric carrier matrix (PEO) [28]. Later on, we have demonstrated for the first time in the literature that chemically modified CD (MβCD) solutions having very high concentration can be effectively electrospun into nanofibers/nanoweb without using any polymeric carrier matrix (Figure 4) [29]. To have deep understanding, we have successfully performed electrospinning of nanofibers from three different chemically modified CDs (HPβCD, HPγCD and MβCD) in three different solvent systems (water, DMF and DMAc) [30]. However, the electrospinning of native CDs (α-CD, β-CD and γ-CD) is more challenging due to their low water solubility when compared to that of chemically modified CDs. Nevertheless, we were able to obtain highly concentrated homogeneous solutions of native CDs by using various solvent systems and we were able to electrospun nanofibers from α-CD [31], β-CD [31] and γ-CD [32]. We have also demonstrated that the electrospun CD nanofibrous webs were also capable for molecular capturing of VOCs (aniline, toluene and benzene) [32-33].

It is well-known that cyclodextrins are used as reducing and stabilizing agent for the synthesis of metallic nanoparticles. In an earlier study, we used HPβCD as an additional reducing and stabilizing agent in order to control size and uniform dispersion of silver nanoparticles (Ag-NP) in electrospun polyvinyl alcohol (PVA) nanofibers [34]. Later on, the green and one-step synthesis of gold nanoparticles (Au-NP) incorporated in electrospun cyclodextrin-nanofibers (CD-NF) without the use of a carrier polymer matrix was also achieved by our group in which CD was used as reducing and stabilizing agent as well as fiber matrix [35]. By using the CD nanofibers as a fiber template, atomic layer deposition (ALD) of metal oxides such as Al_2O_3 and ZnO was performed. Nanocoatings were deposited on polymer-free electrospun cyclodextrin nanofibers in which CD was dissolved in water to obtain nanotubes of Al_2O_3 and ZnO without calcination [36]. CD nanofibers/nanowebs having proper structural stabilization/stability can also surface functionalized with adamantine-containing short-peptide for the purpose of heavy metal removal [37] for waste water treatment or neurite outgrowth [38] for tissue engineering applications.

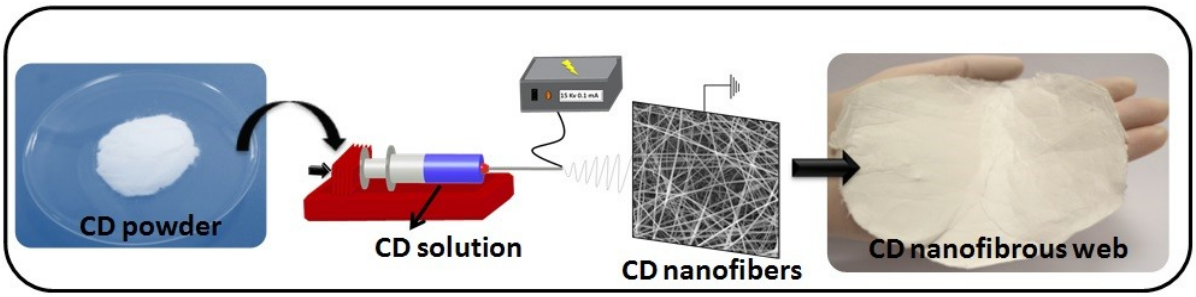

Figure 4. Schematic representation of the electrospinning of pure CD nanofibers without polymeric carrier matrix. The photograph of free-standing electrospun CD nanofibrous web [29-33].

Electrospinning of cyclodextrin-inclusion complex nanofiber

The strategy of incorporation of CD-IC into high surface area polymeric nanofiber matrix may offer practical applications. However, in the case of polymeric matrix used, the loading of the active agents in nanofibers is limited between 3 to 5 % (w/w, with respect to fiber matrix) since incorporation of enhanced amount of CD-IC 50 % (w/w, which corresponds to 3 to 5 % active agent) often creates serious problem for the electrospinning of uniform nanofibers. Additionally, polymer types used could be also a concern about the practices for different applications. So, electrospinning of active agents of CD-IC nanofibers without using a polymer matrix would be quite advantageous due to non-toxic nature of CD with the ability to form inclusion complex which provides high temperature stability, improved water solubility and enhanced antioxidant or antibacterial property. Moreover, much higher loading (10-15 %, w/w) of active agents such as flavor, fragrance and essential oils can be efficiently encapsulated in electrospun polymer-free CD-IC nanofibers than those with polymer matrices and these active agents can be effectively preserved for a longer time due to inclusion complexation.

Recently, we have reported the possibility of electrospinning of polymer-free nanofibers and nanofibrous webs from CD-ICs [39-44]. For the electrospinning of CD-IC by itself without using a carrier polymeric matrix, we find out that the primary criterion is the self-assembly and self-aggregation of these supramolecular CD-IC molecules in their highly concentrated solutions. For instance, free-standing and handy antibacterial electrospun nanofibers/nanowebs from triclosan/cyclodextrin inclusion complexes were successfully produced [39-40]. The electrospinning of UV-responsive supramolecular nanofibers from azobenzene/cyclodextrin inclusion complex was also achieved [41]. The non-toxic and edible nature of CDs make them quite suitable in food applications. Hence, for possible food applications, free-standing and fast-dissolving electrospun nanofibrous webs of geraniol/CD-IC [42], vanillin/CD-IC [43] and limonene/CD-IC [44] were obtained in which these CD-IC nanofibers have shown high thermal stability, enhanced water solubility and enhanced antibacterial and antioxidant properties.

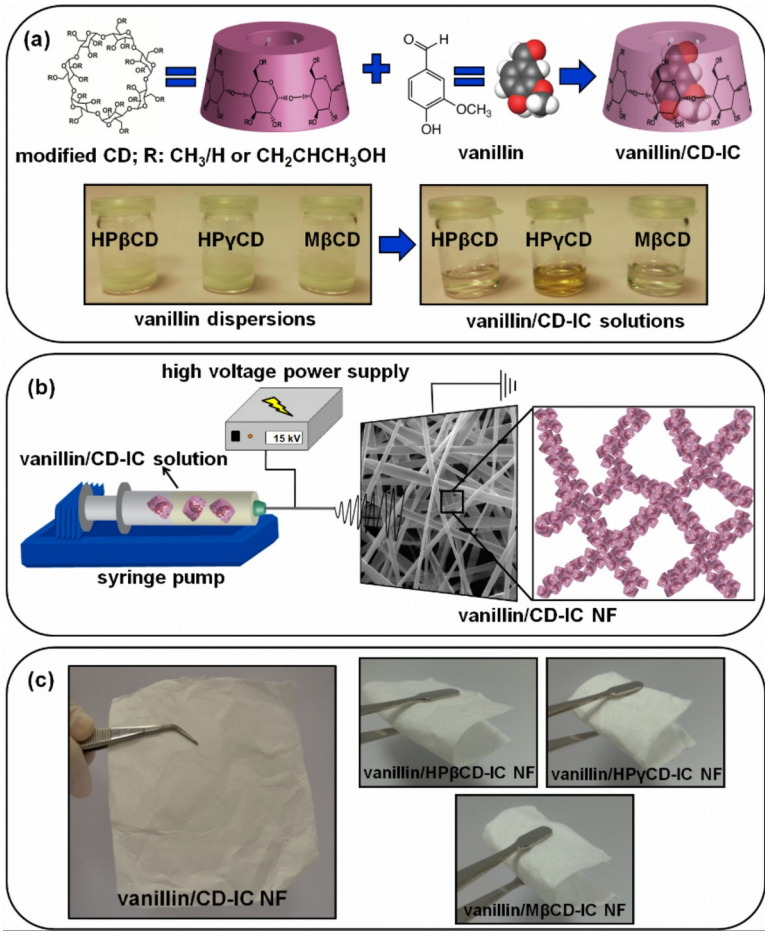

Figure 5. (a) Chemical structure of modified CD and vanillin molecules, schematic representation of IC formation between CD and vanillin, photographs of water based vanillin dispersion and vanillin/CD-IC solutions,
(b) Schematic representation of the electrospinning of vanillin/CD-IC nanofibers,
(c) The photographs of electrospun vanillin/CD-IC nanofibrous webs (figure is taken from [43]).

In brief, the cyclodextrin functionalized electrospun nanofibers would be extremely interesting and also sustainable since such bio-based nanofibrous materials will have unique characteristics having very high specific surface area, high fiber interconnectivity, nano-scale porosity. More importantly, electrospinning of nanofibers having CD functionality and host-guest inclusion complexation capability will enhance/extend the application areas of such nanofibrous materials in environmental/filtration, medical, food, food packaging, textile, cosmetics, agriculture, etc.

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Coplanarity, β-Cyclodextrin solution, Bromocyclohexane

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Radical polymerization, Vinylic supramolecular cross-linkers, γ-Cyclodextrin

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Radical polymerization, Vinylic supramolecular cross-linker, Poly(ethylene glycol)-based terminal-bulky macromonomer, N,N-Dimethyl acrylamide

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Azide-alkin coupling, Host-guest chemistry, Isothermal titration calorimetry, Urea-substituted β-cyclodextrin

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Gel polymer electrolyte, Polyrotaxane, α-Cyclodextrins, Polyethylene glycol, Cross-linked by divinyl sulfone

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Phosphorylation, Non-covalent coating, Iron trimesate, β-Cyclodextrin-based monomers and polymers appended with mannose or rhodamine, Drug delivery

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Characterization, Solubility, Dissolution, Oral bioavailability

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Double loading, HP-β-CD, Nanometric oligolamellar-type vesicles, Photoprotection

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Ketoconazole, Swelling, Drug loading

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Solubility, Freeze-drying, Evaporation, Amorphous form

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Polymeric nanoparticles, Comet assay, Viability, 2-Hydroxypropyl-β-cyclodextrin

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Rifampicin, Isoniazid, Porous particles, Spray-drying, Dry powder inhalers, Inhalational properties

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Inclusion complex and oral delivery system, Epichlorohydrin, Choline chloride, Cationic-β-CD as polymeric core, Alginate and chitosan "petals"

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β-Cyclodextrin, Cytotoxicity against hepatocellular carcinoma (HepG2) cell line, Antibacterial activity, Herbal medicine

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Host-guest complex, Formation constant, Side chain of bisphosphonate

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Inclusion complex, Electrospun, Nanofiber drug delivery systems, Periodontal disease

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Solubilizing agents, Uptake transport, Hydroxypropyl-β-cyclodextrin, Competition assay, Inhibition assay, Inhibitory effects, Undesired drug interactions

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Fluorescence anisotropy, Copper as quencher, Benesi–Hildebrand relationship, Stern-Volmer quenching constant, Hydrophobic interaction

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Complexing agent, Solubilization, Aggregation, Biological membranes, Toxicological profile, Production, Physiochemical properties, Pharmacokinetics, Toxicity

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Nanoparticles, PLGA, Poloxamers, Mesothelioma, Nanoprecipitation

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Inclusion complex, Self-aggregates, Nano-sized particles

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Doxorubicin, Non-covalently connected supramolecular block polymer, Biocompatibility

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2,6-DIMEB, Job's plot, Loading efficiency, Toxicity

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Propylene glycol, HP-β-CD, Glycerol

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Crystallization of curcumin, Elastic modulus, Antioxidant properties, Wound healing ability in hairless mice

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α-CD, β-CD, γ-CD, Surface roughness, Swelling ratio, Improved wound healing in mice

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Anaesthetics, 2-Hydroxypropyl-β-cyclodextrin, Plasma clearance, Mean residence time

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Retention, Stationary phase, Ketoprofen, Fenbufen, Diazepam, m-Xylene

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Osteointegration, Surface morphology, Wettability, Initial burst, Alkaline phosphatase activity, Enhanced bone formation

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Peptide prodrug, Solubility enhancement, Formulation, Hydroxypropyl-β-cyclodextrin, Hydrodynamic radii

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Bioactive compound, Flavonoids

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Modulation of membrane cholesterol content, Methyl-β-cyclodextrin

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Central nervous system, Cholesterol-depleting agents, Magnetic nanoparticles with immobilized cholesterol-depleting agent such as O-methyl-β-cyclodextrin, Triethoxy(3-isocyanatopropyl)silane

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Biodegradable, biocompatible and nontoxic biopolymers, Carriers for in vivo delivery

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Efflux, Pretreatment with β-cyclodextrin, Lowering membrane cholesterol, Hormone

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Gene delivery in living cells, Planar structure

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Cell surface engineering method, Azobenzene-labeled aptamer, Peripheral blood mononuclear cells

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Inclusion complex, Bioactive phytochemicals, Solid-liquid percolation

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Tween-80, Survival of the entrapped cells

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Optimization, Response surface methodology, Duck yolk oil

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Conjugated linoleic acid, Critical micellar concentration, HP-β-CD

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β-Cyclodextrin, Physical properties, Controlled release, Antioxidant food active packaging

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Maltodextrin, β-Cyclodextrin, Whey protein isolate, Gum Arabic, Encapsulation efficiency

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Wine polyphenol, Chokeberry, Blackcurrant, Blueberry, HP-β-CD, Anthocyanin retention during storage, Antiradical activities

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Nutraceutical, Substitution distribution of HP-β-CD

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6. CDs for other Industrial Applications

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Langmuir model, Regeneration experiments, Sorption capacity

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Gas separation, Gas permeation tests, CO_2 permeability, Resistance to physical aging

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Heteropolyacids, Ethyl acetoacetate, α- or β-Naphthol, Benzaldehyde

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Inclusion complex, Carboxymethyl-β-cyclodextrin, TiO_2 surface, Photovoltaic conversion, Photoresponsivity

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N-Isopropylazylamide, Maleic anhydride, Modified β-cyclodextrin, Chiral selector

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