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Cyclodextrins as Bricks for Tuning Polymer Properties

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Abstract

Cyclodextrins are natural cyclic oligosaccharides with a cone shape delimiting a hydrophobic cavity. The rims of cyclodextrins can be functionalized in order to improve their properties. Based on this, cyclodextrins can be linked to polymer chains, which further allows the tuning of the polymer properties. This review describes the methods of polymer functionalization with cyclodextrins and highlights the changes in the physicochemical properties of these materials. This chapter is focused on polymers in solution and in gel states. Cyclodextrin-based polymers are evaluated by various physicochemical methods, such as rheology, calorimetry, and spectroscopy (electron paramagnetic resonance, fluorescence, nuclear magnetic resonance (NMR), Fourier transform infrared (FT-IR), etc.). Both natural and synthetic polymers are considered in this chapter.

Keywords: cyclodextrin-based polymer, synthesis, rheological behavior, EPR spectroscopy

1. Introduction

Cyclodextrins (CDs) are natural compounds, obtained by the enzymatic modification of starch [1], that consist of at least six glucopyranose units linked by α -1,4 glycosidic bond. They have a truncated cone shape delimiting a relatively hydrophobic cavity and two polar rims bearing primary hydroxyl groups (the narrow rim) and secondary hydroxyl groups (the broader rim) [2]. The main feature of CDs is the formation of noncovalent complexes through host-guest interaction, which is, in fact, a summation of several noncovalent steps and overall represents an entropically driven process [3]. The host-guest interactions are determined by different factors, such as size, shape, charge, or polarity of the molecular actors involved [4]. This type of complexation has been extensively documented since the discovery of CDs, especially for low-molecular-weight compounds, by using a variety of physicochemical methods depending on the properties of the guest molecules [5–7]. These methods refer to nuclear magnetic resonance (NMR), UV-Vis, and fluorescence spectroscopies, as well as calorimetric methods. Although these molecules were reported at the end of the 19th century, the explosion of their applications started in the eighth decade of the 20th century [8]. CDs can be involved in the formation

of noncovalent interactions with polymers, giving rise to special types of assemblies known as rotaxanes or pseudorotaxanes [9, 10].

The other feature of CDs is the presence of numerous primary and secondary hydroxyl groups that allow a facile derivatization in order to obtain new molecules that can be used as building blocks for large assemblies. Owing to the difference in reactivity of the primary and secondary groups, it is possible to control the functionalization, which ensures a selectivity of this process. The easiest way to synthesize monoderivatives is by obtaining monotosylated CD, especially for β -CD. This synthesis was studied in detail. A few syntheses are available in order to obtain pure CDs monotosylated at the primary hydroxyl rim that can be used further as bricks for preparing other derivatives. Monotosylated CD can be easily obtained in aqueous alkaline solution in good yields [11]. This derivative will be easily transformed further into amines [12] or thiols [13] that can be modified through maleimides or iodoacetamides [14]. There are also strategies describing the functionalization of primary and secondary rims that can allow obtaining of large supramolecular assemblies. It can be taken into account that the functionalization of the secondary rim is more difficult than the modification of the primary rim, as the hydroxyls that mark the larger rim require a strong base to become activated. In the review of Liu *et al.*, different ways to functionalize CDs at the secondary rims are described [15]. This method has been used to introduce sensing groups that allow studying supramolecular complexes of CDs by electron paramagnetic resonance (EPR) spectroscopy or fluorescence spectroscopy [14, 16]. The applications of these functionalization reactions will be referred in the cases discussed in this chapter.

In this review, we will focus on the two main features of CDs: to generate large supramolecular assemblies through host-guest interactions and to use functionalization in order to improve the properties of polymeric systems by the incorporation of host CD units. The polymeric systems taken into discussion refer to polyrotaxanes and pseudorotaxanes, the particular cases of sliding gels, the pluronic gels, and the functionalization of various synthetic and natural polymers with CD units. An important part will be focused on noncovalent and covalent hydrogels containing CD units.

2. Noncovalent interactions between polymers and cyclodextrins

Rotaxanes are supramolecular structures consisting of at least one ring threaded through an axial molecule, with the particularity that the dissociation of this assembly is hindered by bulky groups at the ends of the axial molecule [17]. CDs may play the role of a ring that is lined up on a polymer chain. The general procedures for the synthesis of polyrotaxanes involve two pathways: the threading followed by rotaxination and the slippage of CD over a bulky group (**Figure 1**). These methods are used for the preparation of CD-based pseudorotaxanes and rotaxanes in aqueous media [18]. The formation of pseudorotaxanes is dependent on the relation between the sizes of the CD cavity and polymeric chain. Thus, α -CD will generate polyrotaxanes and pseudopolyrotaxanes with polyethylene glycols and polyamines [19, 20], while β -CD with polypropylene glycols [21] and γ -CD with polyvinyl alcohol [22], which often leads to gel systems. The endcapping of polyethylene glycols with bulky substituents, such as bis(3,5-dinitrobenzoyl) and bis(2,4-dinitrobenzoyl), will favor the formation of polyrotaxanes with γ -CD [23].

Many water-soluble polymers act as nonionic surfactants by self-association in micelles that are sensitive to temperature and the presence of other molecules. CDs

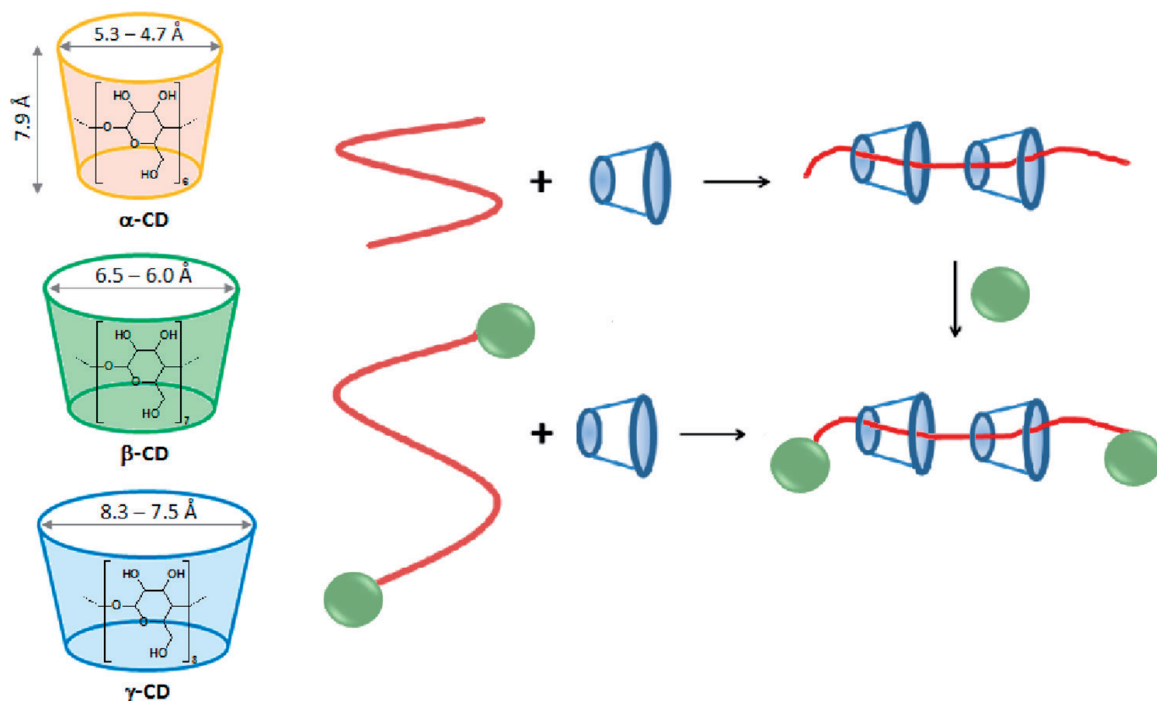


Figure 1.
Schematic representation of CDs and pseudopolyrotaxanes.

can form inclusion complexes with nonionic surfactants from the Triton X or pluronic classes. In the case of Triton X, α -CD can complex the polyethylene chain, while β -CD complexes the nonpolar head (the iso-octylphenyl and phenyl groups) [24]. CDs can also modify the critical micelle concentration or are able to disrupt the micelles due to the complexation of polymers or surfactants [25]. As an example, the Triton X-100 nonionic surfactant, consisting of a short chain of polyethylene glycol and an aromatic nonpolar head, is able to form micelles at concentrations higher than 2.2 mM [26]. CDs can form inclusion complexes with this molecule with different geometries and stoichiometries depending on the CD size. Using isothermal titration calorimetry (ITC) measurements, it was possible to evaluate the binding constants for β -CD and γ -CD by assuming a stoichiometry of 1:1 and 1:2 for β -CD and of 1:1 for γ -CD, both involving the complexation of the nonpolar surfactant head. Conversely, in the case of α -CD, the complexation was supposed to be through the formation of pseudorotaxane by the inclusion of the polyethylene chain, assuming 1:5 stoichiometry, although the ITC data were not conclusive [27]. The interaction of β -CD with Triton X-114 led to the formation of larger aggregates involving hydrogen bonds with β -CD. This has been studied as a function of β -CD concentration and temperature, and it was observed that, at higher β -CD concentration, a transition from micelle to vesicle occurred. This effect is different from the more common effect of CDs on surfactant aggregation [28].

The effects of various CDs on the micellization and gelation of pluronics have also been reported. For such systems, changes in micellar concentration, in gelation, as well as changes of the hydration layer around the polymer chains during phase transition, when CDs are placed among polymeric chains, can be the result of pseudorotaxane formation [29–33]. Being water soluble, CDs will be more probable to target the region of the micelles placed at the water interface. Two studies involving EPR and fluorescence spectroscopies along with rheological and tube inversion methods explore the effect of 2-hydroxypropyl- β -CD on the micelle-to-gel phase transition of pluronic F127 [34, 35]. It was revealed that the spectral parameters of

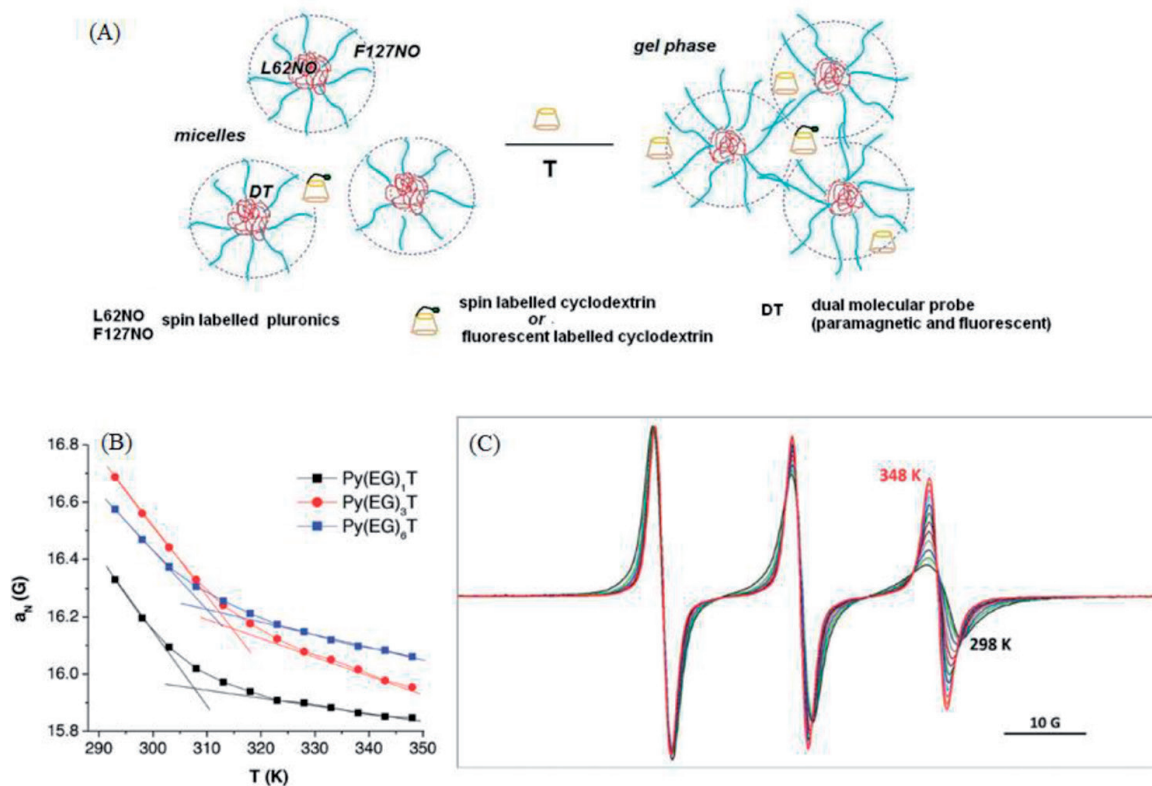


Figure 2. Representation of the micelle-to-gel transition in F127/HPB systems (A), variation of hyperfine splitting constant with temperature (B) and variation of EPR spectra of spin probe with temperature (C) in F127 system [35].

molecular probes (commercially available spin probes, spin-labeled CDs, CDs labeled with fluorophores, or dual molecular probes) deviate from the linear dependence with temperature (**Figure 2**), thus indicating that the macroscopically observed phase transformation is related to changes at the nanoscale level. The results also led to the conclusion that the presence of CDs at the used concentrations does not induce micellar rupture but determines an increase in the micellar water content, which suggests an increase of the micellar size and excludes the formation of pseudopolyrotaxanes.

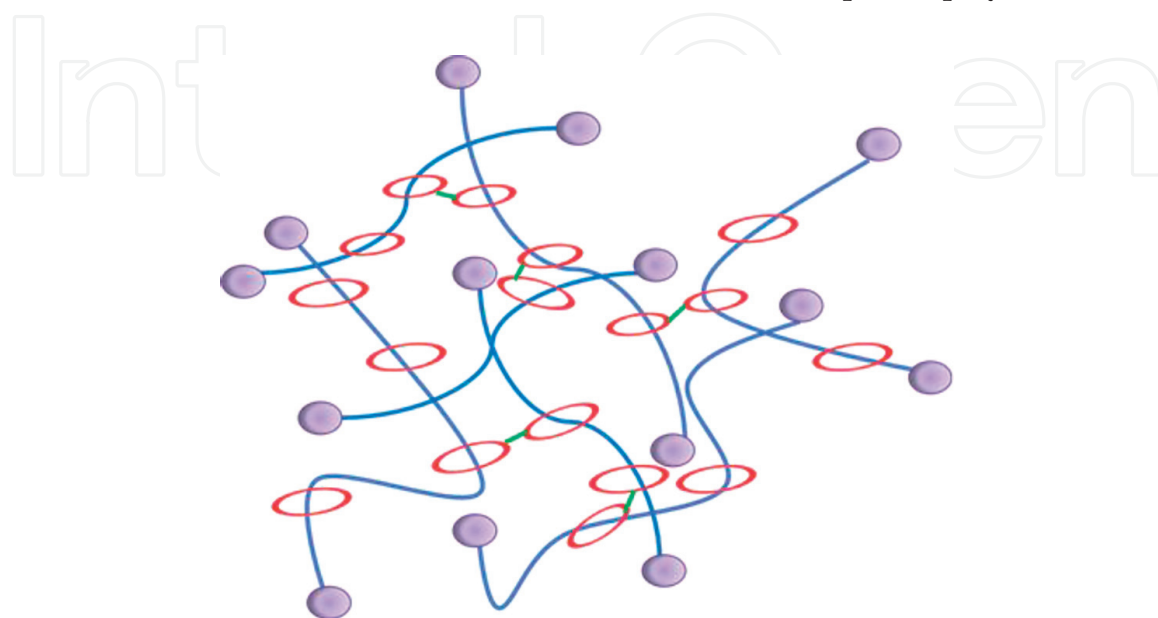


Figure 3. Schematic representation of the supramolecular network of a sliding gel.

Rotaxanes and pseudorotaxanes generate networks that lead to the formation of hydrogels. A particular case refers to the formation of supramolecular networks named sliding gels that are characterized by mobile or sliding crossing points. A classical sliding network results from the intermolecular crosslinking of α -CD/polyethylene glycol pseudorotaxanes (**Figure 3**) [36–38]. The crossing points are, thus, mobile, and this will determine a mobility of the overall network. These gels are formed by linking CD units that belong to different chains.

The properties of these sliding gels can be described by topological parameters such as the complexation degree (number of CD units on a polymer chain), the crosslinker fraction (defined as the ratio of the mole number of crosslinker on the mole number of CDs), and the interactions between the swelling solvent and the constitutive parts of the network [39]. In many cases, the sliding motion depends on the swelling solvent, as well as on other factors such as the pH. For instance, in the case of pseudorotaxanes formed between a triblock copolymer consisting of polyethylene amine/polyethylene glycol/polyethylene amine, and α -CD, with crossing points obtained in the presence of 1,10-carbonyldiimidazole, the gel properties are dependent on the pH value [40].

3. Cyclodextrins covalently attached to polymeric chains

This section aims to summarize new research that has emerged in the past few years on materials composed of synthetic or natural polymers to which CD derivatives have been chemically attached. The polymers discussed here were chosen considering the extensive investigations and applications in pharmaceuticals and biomedicine as drug excipients, biocompatible alternative materials in tissue engineering, contrast enhancers, molecular recognition models, etc. To achieve novel and/or enhanced properties of these classes of compounds, the functionalization with different CD units is employed. To covalently attach CDs onto polymer chains, multiple types of crosslinking agents (citric acid, epichlorohydrin, aldehydes, carbodiimides, and amines) can be used. In recent years, polymers functionalized with CD units have been studied for the development of a variety of polymeric networks [41, 42]. Moreover, the polymeric material based on CD units can be modulated in such a way to form nano/micro/macroparticles, gels, micelles, coating films, or fibers [43–45]. Many studies in this regard are performed using chitosan, mostly due to its remarkable biological properties, including antimicrobial activity, nontoxicity, biocompatibility, and biodegradability, and at the same time to the possibility to functionalize it [46, 47].

For instance, Campos *et al.* took this advantage and appended β -CD on chitosan nanoparticles in the presence of N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide (EDC) and N-hydroxysuccinimide (NHS), thus developing a potential carrier for botanical pesticides [48]. A green-assembly strategy to obtain β -CD-chitosan functionalized graphene oxide hydrogels in the presence of sodium ascorbate has been reported, with application as recyclable decontaminants in wastewater treatment [49]. Both simple polymers and polymer mixtures are favorable to the attachment of CD units, as demonstrated by the research of Hardy *et al.* regarding the complex formed between chitosan and alginate for the release of piroxicam [50]. The research revealed that CD appending on chitosan generates a decrease in the number of amino groups, thus modulating the alginate complexation. In a recent study, alginate has also been functionalized with either host units (n-alkyl amine CD derivatives) or guest units (adamantane) [42]. In this particular case, the grafting procedure was

performed in aqueous media in the presence of EDC and N-hydroxysulfosuccinimide (NHSS). The properties of the gel obtained by mixing the two types of alginates in the presence of Ca^{2+} ions were influenced by the host-guest interaction and the length of the alkyl chain of the β -CD derivatives. Thus, by mixing the decorated alginates with CD and adamantyl units, materials suitable for encapsulating both large molecules and small species can be obtained. In addition, the functionalization of alginate with units of β -CD derivatives by using Cu(I)-catalyzed azide-alkyne cycloaddition click reaction led to materials with good drug release properties [51]. The same procedure was used in another study, showing that the grafting degree of β -CD on alginate can be controlled and modulates the release/uptake of the model molecule methyl orange [52]. In addition, a composite based on sodium alginate grafted with β -CD using epichlorohydrin and NaOH was successfully obtained for the first time and used as a matrix in the immobilization of *Arthrobacter simplex* cells for cortisone acetate biotransformation [53].

Not only natural polymers were grafted with CD derivatives, but also synthetic ones. In this regard, the polyacrylic acid was modified by attaching a CD derivative (2-aminoethyl)amino-deoxy- β -CD) [54]. The study targeted to obtain the self-assembly of β -CD and adamantyl moieties covalently linked to polyacrylate networks for application in controlled complexation and release of ethyl orange, methyl orange, and methyl red. Another study developed a multistimulus responsive supramolecular hydrogel based on host-guest and electrostatic interactions between β -CD dimer and methoxy-azobenzene molecules grafted on polyacrylic acid [55]. The obtained materials showed thermo-, photo-, and pH-responsive behavior determining a reversible sol-gel transition.

Chabalala *et al.* described the grafting of β -CD molecules on polyacrylonitrile using citric and sulfuric acids as crosslinkers [56]. The nanofiber membranes produced by the electrospinning method were used for the adsorption of bromophenol blue and atrazine. Mono-(6-ethylenediamine-6-deoxy)- β -CD was appended in the presence of EDC/NHS crosslinking agents onto the external surface of a plasma separation membrane based on polyvinylidene fluoride [57].

4. Polymer gels containing cyclodextrins

Polymers containing CDs have the ability to form supramolecular hydrogels mostly due to host-guest complexation or by the inclusion of linear polymeric chains into host cavities [58]. The latter leads to the formation of pseudorotaxanes. Literature data show that, unlike β -CD, α and γ -CD are able to generate interactions favorable to the obtaining pseudorotaxanes [59]. However, in particular cases, pseudorotaxanes were prepared using β -CD and polylactic acid or polypropylene glycol [60, 61].

Several studies regarding host-guest interactions in hydrogel systems are reported in the literature [60, 62, 63]. By appending host and guest units to the polymer chains, one can modify and control their behavior in solutions and obtain gel systems. This strategy ensures conditions for the creation of topological crosslinks based on host-guest interactions, which have the advantage of being reversible and movable. Host and guest units can be grafted on the chains of numerous polymers (hyaluronic acid [64, 65], carboxymethyl cellulose [66, 67], sodium alginate [42, 68], polyacrylic acid [55, 69, 70], polyvinyl alcohol [71, 72], polymethyl vinyl ether-alt-maleic acid [73, 74], poly-N-isopropylacrylamide [75], and polyethylene glycol [76]). These supramolecular gels find use in the medical field for drug delivery, tissue culture, and

medical treatments [77, 78]. Depending on the desired application, hydrogels formed by host-guest interactions can be generated in several ways. Therefore, CD having the role of guest molecules can be grafted separately on polymer chains and mixed with polymers bearing guest grafts to form supramolecular assemblies [41, 60].

The formation of alginate gels in the presence of divalent cations was monitored by EPR spectroscopy considering the changes in the dynamics of spin-labeled alginate chain [79]. In a recent study, we showed that the functionalization of alginate with CD (as host) or adamantane (as guest) influences the properties of ionotropic generated gel in the presence of Ca^{2+} ions [42]. As a consequence, polymer functionalization and subsequent interactions between the appended host and guest units change the morphology of the resulting xerogels (**Figure 4**).

In fact, the derivatization, together with the host-guest interactions, has an impact on the rheological properties, i.e., the hydrogels made from a mixture of adamantane-functionalized and CD-functionalized alginates presented higher storage and elastic

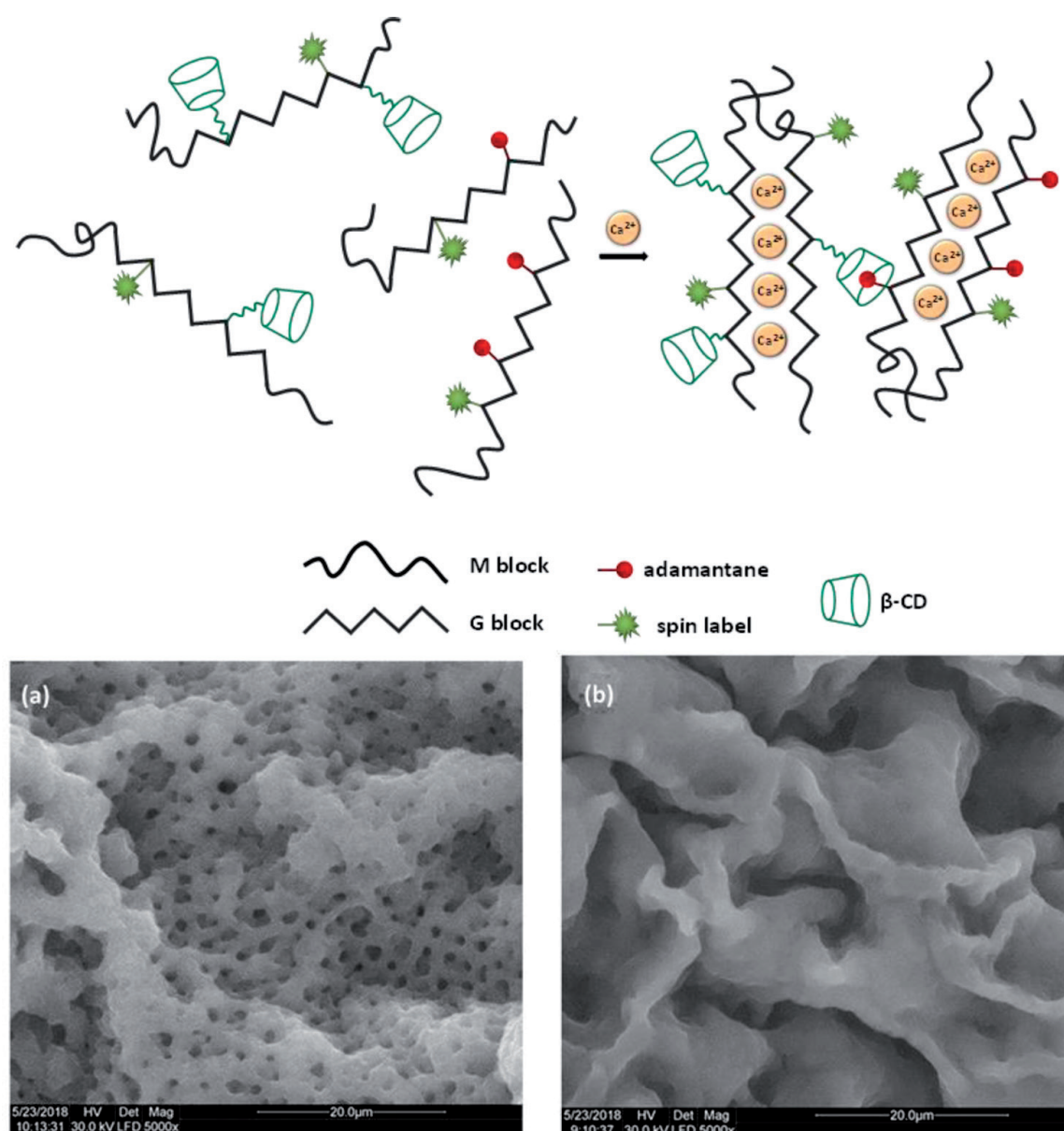


Figure 4. Schematic representation of ionotropic gelation of functionalized alginates. SEM images of alginate xerogels: (a) nonfunctionalized alginate and (b) alginate functionalized with CD and adamantane units [42].

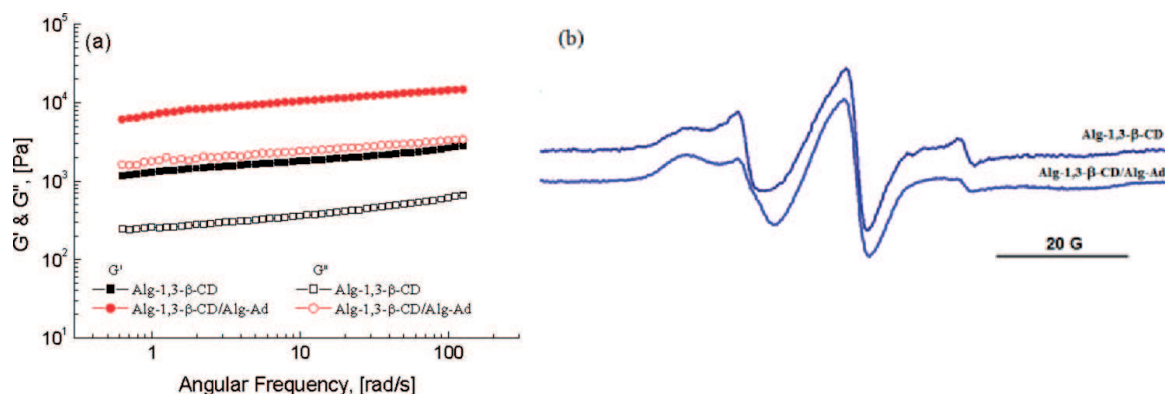


Figure 5. (a) Viscoelastic properties and (b) EPR spectra of alginate hydrogels functionalized with β -CD or adamantane units [42].

moduli values (**Figure 5a**). Appending the paramagnetic moieties to these functionalized alginates allows evidencing the changes at the molecular level of the dynamic of the overall motion (**Figure 5b**). All these findings led to the conclusion that the presence of host-guest interactions can modulate the features of alginate hydrogels.

The polymerization of the inclusion complex is another technique that can be used to obtain CD-based hydrogels. Ikura *et al.* approached the free-radical copolymerization of polyethyl acrylate crosslinked with peracetylated γ -cyclodextrin methylacrylamide monomer and acrylate monomers and investigated the effect of the size of the main chain monomers on the formation of movable crosslinking points [80]. Thus, it was observed that the small polymer main chains penetrated the CD units and acquired the role of movable crosslinking points in the hydrogel, whereas copolymerization with bulky monomers leads to hydrogels without movable crosslinking points.

In aqueous media, host-capped polymers can be mixed with guest-grafted chains to obtain hydrogels. Ioniță *et al.* showed that the reaction of isocyanate end-capped polyethylene glycol with β -CD leads to the formation of a fibrous gel with covalent network [81]. Moreover, by adding spin probes (e.g., TEMPO and adamantane-TEMPO), it was possible to determine the extent by which gel fibers were affected by hydrogen bonding interactions with solvent, crosslinks density, or temperature. The study revealed that, at low temperature, ice crystallization is prevented inside the gels, and this phenomenon is accompanied by the formation of supercooled water.

Another method to obtain hydrogels is the mixing of end-capped guest crosslinkers with certain host-grafted polymers [41, 60]. A recent study has shown that the multivalence effect within a polyethylene glycol-adamantane/ β -CD-alginate system can be quantified to create hydrogel-like cell matrices [82]. The complexation of CD functionalized alginate with adamantyl end groups on polyethylene glycol chains changed the valence of the system. Thus, a correlation could be observed between the multivalence generated by the variation in the number of polyethylene glycol arms and the strength of binding affinities inside the hydrogel.

A particular type of gel is formed between guest-grafted polymers that are capable of forming multilayer vesicles and small host molecules grafted on different polymeric chains. A good example is the gel formed by thiolated monolith polymers, in which β -CD vesicles were introduced in order to formulate a hydrogel with pH-responsive properties [83].

Polymeric gel formation can be described using rheological, viscosity, and dynamic light scattering measurements. These methods provide global information

on such systems. Spectroscopic methods, such as fluorescence, IR, or UV-Vis spectroscopy, are often used to describe changes in the organization of macromolecules that are usually governed by noncovalent interactions [84]. In the particular case of gel formation, electron microscopy techniques are used to evidence the gel fibers. An interesting, powerful, but still rarely used approach in studying gels involves EPR spectroscopy [34, 79, 81, 84, 85]. The EPR spectroscopy is suitable to study polymer systems and gels as the method can provide insights into local, static, and dynamic properties of these systems. This method can evidence nanoscale inhomogeneities in polymers systems [86, 87].

By using spin-labeled CDs, it was possible to monitor the gel formation process, while the diffusion of various spin probes can evidence the nonuniform properties of covalent gels [81, 88, 89]. Other EPR studies explored the self-assembly of pluronic F127 leading to gel phase as a function of temperature and concentration of CD [34, 35] or the formation of supramolecular gels resulted by the assembly of low-molecular-weight gelators [85].

5. Applications

Over the past four years, data from the literature indicate some comprehensive reviews of CD-based polymer applications [63, 90–93]. The most common applications of these materials are in the field of drug delivery [60, 94, 95], biomedical engineering [90, 96, 97], food industry [93, 98, 99], responsive adhesives [100, 101], coatings [102, 103], sensors [104, 105], and environmental remediation [106, 107].

Table 1 exemplifies some of the most recent applications reported in the literature on polymers functionalized with CD units.

Material	Applications	Ref.
β -CD-chitosan nanoparticles	Carrier for carvacrol and linalool	[48]
β -CD-chitosan/graphene oxide hydrogel	Removal of methylene blue	[49]
β -CD-chitosan/alginate complex	Encapsulation and release of piroxicam	[50]
β -CD-alginate hydrogels	Paclitaxel drug release	[51]
β -CD-alginate gel beads	Release of methyl orange	[52]
β -CD-alginate	Matrix for <i>Arthrobacter simplex</i> cell immobilization	[53]
β -CD-polyacrylonitrile nanofiber	Adsorption of bromophenol blue and atrazine from aqueous systems	[56]
β -CD polymer-tetrafluoroterephthalonitrile	Removal of malachite green	[108]
β -CD polymer-tetrafluoroterephthalonitrile	Agent for monitoring endocrine disrupting chemicals from water	[109]
β -CD based polymeric adsorbent	Pollutants removal (rhodamine B, Congo red and cadmium ions) from wastewater	[110]
2-hydroxypropyl- β -CD-polyacrylic acid	Removal of ibuprofen	[111]
β -CD-activated charcoal-Na alginate magnetic beads	Removal of methyl violet, brilliant green, norfloxacin, ciprofloxacin and copper ions from aqueous systems	[112]

Material	Applications	Ref.
β -CD-carboxymethyl chitosan	Sensor for direct determination of manganese	[113]
β -CD-alginate-graphene oxide hydrogel	Injectable hydrogel for soft tissue engineering	[114]
β -CD-epichlorohydrin-carboxymethyl chitosan	Bioactive enhancement of cyanidin-3-glucoside	[115]
β -CD-polyurethane/chitosan	Gentamicin sulphate drug release	[116]
β -CD-Fe ₃ O ₄ -chitosan nanoparticles	Support for lipase immobilization	[117]
β -CD-chitosan beads	Support for keratinase immobilization	[118]

Table 1.
Applications of polymers functionalized with β -cyclodextrin.

6. Conclusions and perspectives

The general lines of polymer functionalization with cyclodextrins and resulting changes of their properties have been reviewed in this chapter. These can be applied for generating and studying other polymer-cyclodextrin systems. The EPR spectroscopy can be used as a method for proving changes at nanoscale level in such systems due to host-guest interactions occurring in polymer systems. The EPR data can be linked with other experimental data provided by classical methods used to characterize polymers in solution and in gel states, like rheology, electron microscopy techniques, etc.

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Conflict of interest

The authors declare no conflict of interest.

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