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Polymer-free cyclodextrin and natural polymer-cyclodextrin electrospun nanofibers: A comprehensive review on current applications and future perspectives

Andrea Dodero, Guy Schlatter, Anne Hebraud, Silvia Vicini, Maila Castellano

► To cite this version:

Andrea Dodero, Guy Schlatter, Anne Hebraud, Silvia Vicini, Maila Castellano. Polymer-free cyclodextrin and natural polymer-cyclodextrin electrospun nanofibers: A comprehensive review on current applications and future perspectives. *Carbohydrate Polymers*, 2021, 264, pp.118042. 10.1016/j.carbpol.2021.118042 . hal-03217583

HAL Id: hal-03217583

<https://hal.science/hal-03217583>

Submitted on 4 May 2021

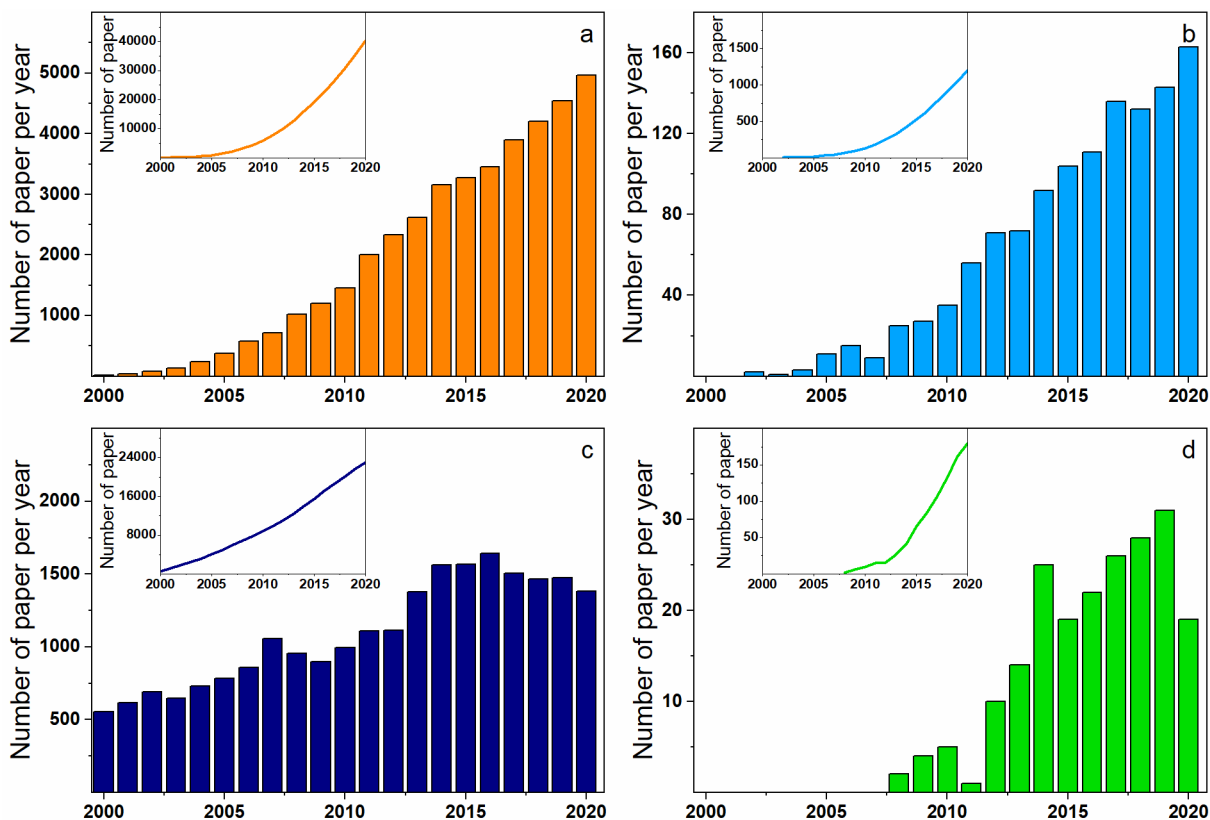
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- 28 Electrospinning; Cyclodextrins; Natural biopolymers; Functionalized nanofibers; Inclusion complexes;
- 29 Polycyclodextrins.

30 **1 Introduction**

31 Electrospinning is a simple, versatile, and cost-effective technique that allows the fabrication of polymeric
32 nano- and microfibers starting from a polymer solution or polymer melt (J. Xue et al., 2019). Demonstrated
33 in 1934 by A. Formhals (Anton, 1934), electrospinning started to gain considerable interest from the 2000s
34 and it is nowadays one of the most employed fiber-forming approaches for several purposes (Ahmed et al.,
35 2015; Dodero, Alloisio, Castellano, et al., 2020; Dodero, Brunengo, Castellano, et al., 2020; Dodero, Vicini,
36 Lova, et al., 2020; Tebyetekerwa & Ramakrishna, 2020; Thenmozhi et al., 2017). Among others, electrospun
37 nanofibers can be efficiently used to encapsulate a great variety of molecules, such as drugs, bioactive
38 compounds and nanoparticles (Castellano et al., 2019; Figueiredo et al., 2020; Quirós et al., 2016; Torres-
39 Giner et al., 2016; Wen et al., 2017). In this sense, cyclodextrins (CDs) offer the chance to obtain
40 functionalized nanofibers with high encapsulation efficiency, enhanced thermal stability, water-solubility,
41 and bioavailability of the encapsulated molecules (Costoya et al., 2017; Senthil Muthu Kumar et al., 2019;
42 Topuz & Uyar, 2019). However, cyclodextrin-functionalized nanofibers remain a barely explored research
43 field and, to the best of our knowledge, no commercial products currently exist besides an increasing number
44 of original research work has being recently published. To this regard, Costoya et al. (Costoya et al., 2017)
45 recently reviewed the fabrication of cyclodextrin functional nanofibers, whereas Topuz et al. (Topuz & Uyar,
46 2019) and Vidal et al. (Patiño Vidal et al., 2021) focused the attention on their pharmaceutical and food
47 packaging applications, respectively. By way of example, Figure 1 reports the number of publications per
48 year in the last two decades (i.e., 2000-2020) concerning electrospinning (Figure 1a), functionalized
49 nanofibrous membranes (Figure 1b), cyclodextrins (Figure 1c) and cyclodextrin-functionalized nanofibrous
50 membranes (Figure 1d).

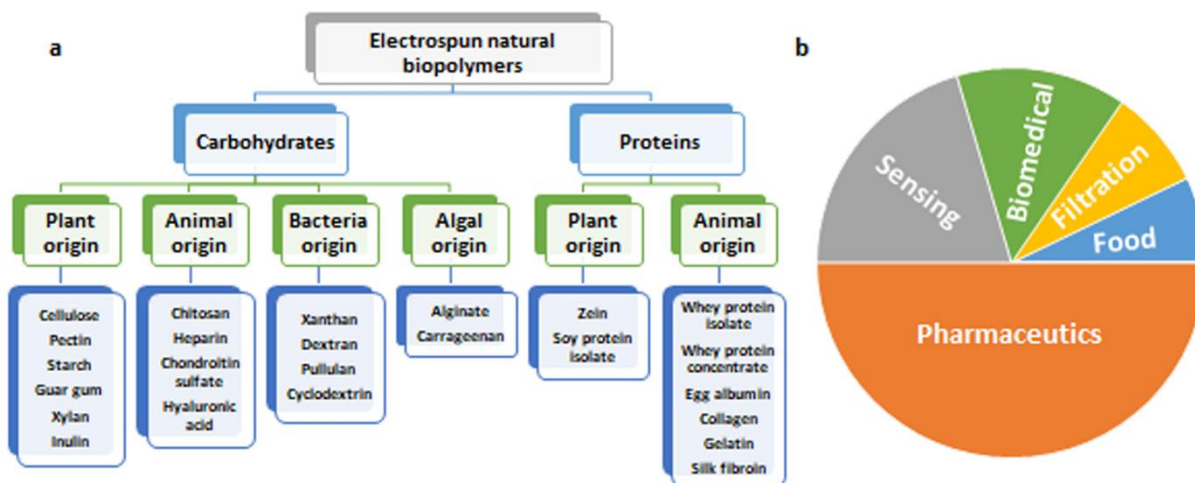


51

52 **Figure 1.** Annual trend of publications regarding (a) electrospinning, (b) functionalization of electrospun
 53 nanofibers, (c) cyclodextrins, and (d) cyclodextrin-functionalized electrospun nanofibers. Insets represent the
 54 cumulative number of publications. Data has been retrieved from Scopus database.

55

56 Among the great variety of commercial polymers that can be electrospun, naturally occurring materials
 57 represent an attractive alternative to synthetic ones (Ding et al., 2019; Soares et al., 2018). Indeed,
 58 biopolymers, such as polysaccharides and proteins, can be used to fabricate nanofibers with superior
 59 biocompatibility, biodegradability, and lower environmental impact compared to industrially derived
 60 materials. Nonetheless, their processing is usually more difficult and it requires a series of experimental
 61 precautions (Bertasa et al., 2020; Ding et al., 2019; Dodero, Pianella, et al., 2019; Kakoria & Sinha-Ray,
 62 2018; Mendes et al., 2017; Noruzi, 2016). A schematic representation of the most common natural
 63 biomaterial-based electrospun mats and their applications is reported in Figure 2.



64

65 **Figure 2.** (a) Schematic of natural biopolymer-based electrospun membranes depending on the source
 66 materials and (b) pie chart representing their main applications according to the Scopus database.

67

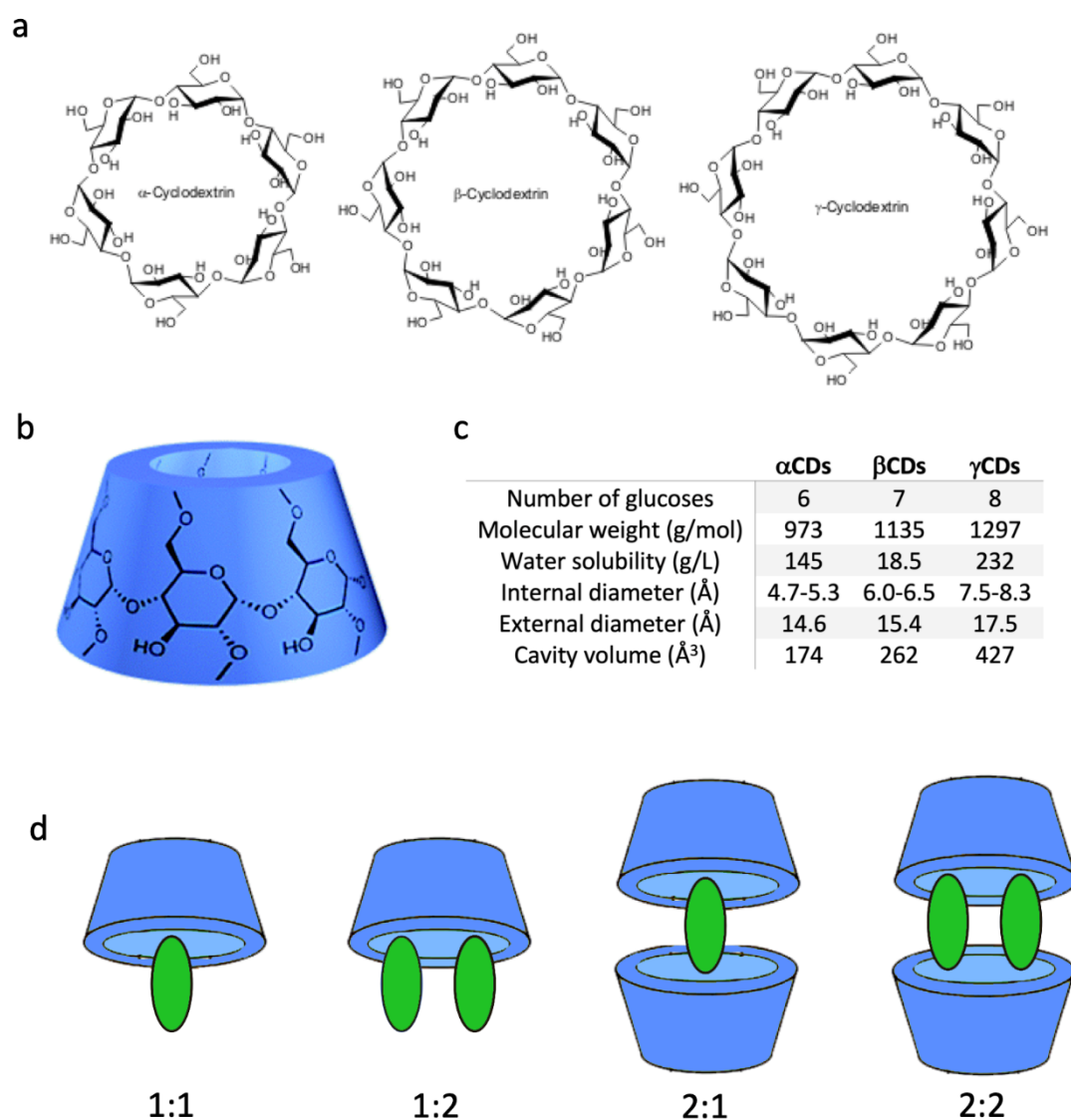
68 With these premises, aiming to highlight the importance of developing environmentally benign materials,
 69 this review aims to provide a comprehensive summary of the recent advances in the preparation of either
 70 polymer-free cyclodextrin-based nanofibers, polycyclodextrin-based nanofibers, or natural polymer-based
 71 (i.e., polysaccharides and proteins) nanofibers functionalized with cyclodextrins. To this end, after a brief
 72 introduction concerning cyclodextrins first and electrospinning then, a critical and complete overview of the
 73 current literature is presented and discussed. Finally, a short consideration about the future trends and
 74 possibilities is reported together with a conclusion paragraph.

75

76 2 Cyclodextrins

77 2.1 Structure and Properties

78 Cyclodextrins consist of cyclic oligosaccharides of glucopyranose assuming the structure of a hollow
 79 truncated cone (Figure 3b) (Sá Couto et al., 2015). The inner cavity of this three-dimensional structure is
 80 partially hydrophobic owing to the -CH and -CH₂ carbons and ether oxygens, whereas the exterior surface is
 81 hydrophilic due to the high number of hydroxyl groups (Szejtli, 1998). Although several types of CDs exist,
 82 the most common ones consist of 6, 7, and 8 glucopyranose units and are named α CDs, β CDs, and γ CDs,
 83 respectively, with their structure and main properties reported in Figure 3a-c (Del Valle, 2004; Jansook et al.,
 84 2018; Kurkov & Loftsson, 2013).



85

86 **Figure 3.** (a) Chemical structure of the most common CDs. (b) Three-dimensional conformation taken by
 87 CDs. (c) Summary of the main physical properties of CDs depending on the glucose unit number. (d)
 88 Schematics of inclusion complex formation with blue and green objects representing cyclodextrins (i.e., host
 89 molecules) and small molecules (i.e., guest molecules). For example, inclusion complexes with a molar ratio
 90 1:1 have been prepared between β CDs and racecadotril (Semalty et al., 2014), with a molar ratio 1:2 between
 91 β CDs and naproxen (Grandelli et al., 2013), with a molar ratio of 2:1 between β CDs and carvacrol (Bethanis
 92 et al., 2013), and with a molar ratio 2:2 between β CDs and linalool (Ceborska, 2016).

93

94 Native CDs are highly crystalline and are known to adopt a cage structure with a herringbone arrangement
 95 (Saenger et al., 1998). Typically, they present a low solubility that depends on their size, with β CDs being
 96 the less soluble ones (Szejtli, 1998). In order to overcome this processing limitation, CDs have been
 97 chemically modified to enhance their water solubility mostly by causing a considerable loss of crystallization.
 98 Among others, the most studied are randomly methylated- β CDs (M β CDs), 2-hydroxypropyl- β CDs

99 (HP β CDs) and 2-hydroxypropyl- γ CDs (HP γ CDs) (Crini, 2014; Landy et al., 2012). Remarkably, an
100 interesting feature of native and modified CDs is represented by their capability to undergo
101 crosslinking/polymerization reactions leading to water-insoluble cyclodextrin-based polymers (polyCDs)
102 with tailor-made properties (Blanchemain et al., 2008; Crini, 2014; Morin-Crini & Crini, 2013; Sherje et al.,
103 2017).

104

105 **2.2 Cyclodextrin Inclusion Complexes**

106 Cyclodextrins are nowadays applied in many industrial sectors (Q. Liu et al., 2020a; Matencio et al., 2020;
107 Wankar et al., 2020; Ying - Ming Zhang et al., 2020). The main reason for CDs success is related to their
108 unique ability to form non-covalent host-guest complexes with a variety of small molecules via inclusion in
109 their hydrophobic cavity (G. Chen & Jiang, 2011; Folch-Cano et al., 2014; Mura, 2015). Depending on both
110 the type of cyclodextrins and hydrophobic molecules, as well as on the formulation conditions, the
111 complexation can occur between a host and a guest molecule, a host and two guest molecules, or two hosts
112 and a guest molecule as represented in Figure 3d (Frömming & Szejtli, 1994; Kim et al., 2006; S. Li & Purdy,
113 1992; Mohanty et al., 2006; Song et al., 2009). Cyclodextrin inclusion complexes (CD-ICs) are commonly
114 exploited to protect those molecules that are sensitive to light, oxygen, heat, and chemical reactions, as well
115 as to increase the water solubility of hydrophobic compounds and to preserve highly volatile
116 fragrances/flavours. By way of examples, CD-ICs have been widely proposed in food and beverage products
117 (dos Santos et al., 2017; É. Fenyvesi et al., 2016), in the pharmaceutical and (Jansook et al., 2018) cosmetic
118 fields (Crini et al., 2018), in the textile industry (Narayanan et al., 2015; Singh & Sahu, 2018), and in
119 developing filtering materials (Éva Fenyvesi et al., 2020; Q. Liu et al., 2020b).

120

121 **3 Electrospinning and its Use as Encapsulation Technique**

122 The electrospinning (ES) technique consists of the application of a high voltage to a polymer solution, or a
123 polymer melt, that is usually extruded from a spinneret at a constant rate. Owing to the presence of free
124 surface charges, the polymeric droplet exiting the spinneret takes the shape of a cone (i.e., Taylor cone) and,
125 when the repulsive electrostatic forces overcome the liquid surface tension, a continuous jet erupts from the

126 spinneret tip. Hence, solid nano- or microfibers are obtained and generally collected in a pseudo-random
127 manner on a grounded collector (Lukáš et al., 2009). The stability of the polymer jet and the related fiber
128 morphology depend on the spinning system properties, processing parameters and environmental conditions
129 (Amiraliyan et al., 2009; Ismail et al., 2017; Thompson et al., 2007). In this sense, electrospinning represents
130 an easy, versatile, and industrially scalable approach to prepare fibers with tailored properties (Kannan et al.,
131 2016; J. Xue et al., 2019; M. Yu et al., 2017).

132 During the past decades, both synthetic and naturally derived polymers have been electrospun. Synthetic
133 polymers are usually easier to electrospun and allow good reproducibility and superior mechanical
134 performances. Additionally, by using hydrophobic materials, water-resistant mats able to maintain their
135 peculiar structure in contact with aqueous media can be easily obtained without the need for post-production
136 treatments. However, such materials usually require the use of organic hazardous solvents to be processed
137 thereby representing a substantial environmental and health threat. Conversely, the electrospinning of
138 naturally occurring polymers is starting to be broadly investigated as a less impacting and “greener”
139 alternative. Indeed, besides the fact that they can be processed via eco-friendly solvents (e.g., water, slightly
140 acid mixtures, etc), natural biopolymers can be often extracted from food and agricultural wastes hence
141 endowing the interesting possibility to develop circular economy products (Ding et al., 2019; Kakoria &
142 Sinha-Ray, 2018; Mendes et al., 2017; Noruzi, 2016; Schiffman & Schauer, 2008). Moreover, such materials
143 are usually biocompatible, weakly immunogenic, biodegradable, and digestible, and present other peculiar
144 properties that make them extremely promising for the fabrication of biomedical and pharmaceutical
145 products, as well as for food packaging and biosensor applications (Dodero, Lova, Vicini, Castellano, et al.,
146 2020; Galiano et al., 2018). Nevertheless, natural polymers present drawbacks such as poor reproducibility,
147 mechanical properties and thermal resistance, and usually require a post-processing crosslinking reaction to
148 exhibit appropriate stability and performances. Hence, it is not surprising that the electrospinning of such
149 materials is considerably harder compared to synthetic polymers (Valencia et al., 2019; Yi et al., 2020).

150 With these premises, it is not surprising that electrospinning is nowadays widely used to prepare nanofibrous
151 membranes for a broad range of purposes. For example, electrospun mats find several uses as biomedical and
152 pharmaceutical devices (Cheng et al., 2018; Miguel et al., 2018), in filter materials (D. Lv et al., 2018;
153 Snowdon & Liang, 2020), in the packaging sector (Senthil Muthu Kumar et al., 2019; C. Zhang et al., 2020),

154 but also for energy-related applications (J. Wang et al., 2020), sensors and biosensors (Han et al., 2019;
155 Rozemarie Manea & Berteau, 2019), and textile products (Mirjalili & Zohoori, 2016). Additionally,
156 electrospinning has proved to be a promising encapsulation technique with several advantages compared to
157 the common industrial approaches, such as spray-drying and solvent evaporation (Becerril et al., 2020; Celli
158 et al., 2015; Lohith Kumar & Sarkar, 2018; Shishir et al., 2018). Specifically, apart from its versatility, the
159 key advantage of electrospinning as an encapsulation technique consists of the absence of heat, which is
160 fundamental to preserve the structure and properties of the entrapped molecules. However, most of the drugs
161 and bioactive compounds of major interest are characterized either by low water solubility, poor thermal
162 stability or a highly volatile nature. Hence, to overcome such limitations and improve the efficiency of
163 electrospinning as an encapsulation technique, the use of cyclodextrins to obtain functionalized nanofibers
164 has recently gained increasing relevance.

165

166 **4 Polymer-free Electrospun Cyclodextrin – based Membranes**

167 One of the main requirements to obtain defect-free homogenous nanofibers via electrospinning technique is
168 the presence of sufficient intermolecular interactions in the working solutions (or melts). In polymer-based
169 systems, these are usually provided by chain entanglement (Mendes et al., 2017; C. Wang et al., 2016). On
170 the contrary, the electrospinning of low molecular weight molecules is quite a challenge because of the
171 complete lack of entanglements. Hence, only a few can be electrospun alone. Among others, owing to their
172 unique self-assembling behaviour, pure phospholipids (McKee et al., 2006), surfactants (Cashion et al.,
173 2010), tannic acid (Allais et al., 2018), and cyclodextrins (Celebioglu & Uyar, 2010) can be used to prepare
174 polymer-free nanofibers. In this sense, thanks to their unique physical-chemical properties, polymer-free
175 cyclodextrin nanofibers have gained huge interest in recent years. However, their proficient usage in
176 industrial applications remains a challenge and further studies are required to obtain products with effective
177 large-scale applicability (Costoya et al., 2017; Topuz & Uyar, 2019, 2020). In the following, a detailed
178 summary (see Table 1) of the current state-of-the-art is provided by reviewing both proofs of concept and
179 applicative reports.

180 **Table 1.** Comprehensive summary of polymer-free cyclodextrin electrospun membranes and their main applications. If not indicated (---), the used solvent is only water.

Application	Cyclodextrin	Encapsulated compounds	Solvent	Ref.
Fundamental studies	M β CDs	---	Water, DMF, DMAc, Urea	(Celebioglu & Uyar, 2010, 2012)
	HP β CDs	---	Water, DMF, Urea	(Manasco et al., 2012; W. Zhang et al., 2012)
	HP γ CDs	---	Water, DMF, DMAc, Urea	(Celebioglu & Uyar, 2012)
	α CDs	---	Water, DMF, DMAc, DMSO, water – NaOH	(Celebioglu & Uyar, 2013a)
	β CDs	---	Water, DMF, DMAc, DMSO, water – NaOH, ionic liquids	(Ahn et al., 2013; Celebioglu & Uyar, 2013a)
	HP α CDs	---	Water, tap water, salty water	(Celebioglu & Uyar, 2020b)
Drug and bioactive compound delivery systems	HP β CDs	Triclosan	---	(Celebioglu, Umu, et al., 2014; Celebioglu & Uyar, 2011)
		Azobenzene	---	(M. Chen et al., 2013)
		Geraniol	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Tekinay, et al., 2016)
		Camphor	---	(Celebioglu, Aytac, et al., 2018)
	HP γ CDs	Triclosan	---	(Celebioglu, Umu, et al., 2014; Celebioglu & Uyar, 2011)
		Geraniol	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Tekinay, et al., 2016)
		Camphor	---	(Celebioglu, Aytac, et al., 2018)
	M β CDs	Geraniol	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Tekinay, et al., 2016)
Fast-dissolving nanofibers for the rapid compound administration	HP β CDs	Spironolactone	EtOH	(Vigh et al., 2013)
		Diclofenac	EtOH	(Balogh et al., 2015)

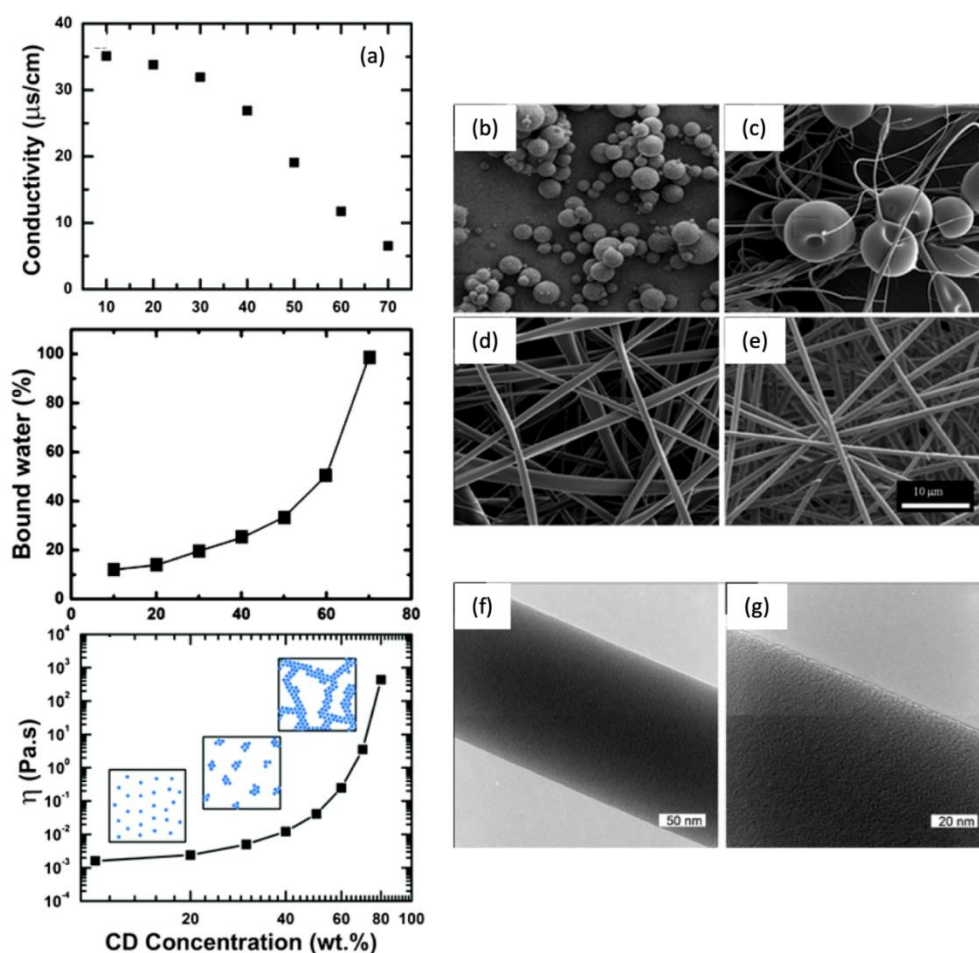
sodium		
Vitamin E	Water – EtOH	(Celebioglu & Uyar, 2017)
Paracetamol	---	(Zehra Irem Yildiz & Uyar, 2019)
α -lipoic acid	---	(Celebioglu & Uyar, 2019a)
Ibuprofen	---	(Celebioglu & Uyar, 2019b)
Metronidazole	---	(Celebioglu & Uyar, 2019c)
Curcumin	---	(Celebioglu & Uyar, 2020c)
Hydrocortisone	---	(Celebioglu & Uyar, 2020d)
Ferulic acid	---	(Celebioglu & Uyar, 2020e)
β -galactosidase	---	(Vass et al., 2020)
Doxycycline	---	(Kiss et al., 2020)
Thiabendazole	---	(Gao et al., 2020)
Vitamin-A acetate	---	(Celebioglu & Uyar, 2020a)
Vanillin	Water, DMF, DMAc	(Celebioglu, Kayaci-Senirmak, et al., 2016)
Limonene	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Kusku, et al., 2016)
Linalool	---	(Aytac, Yildiz, et al., 2017)
Citral	---	(Aytac, Celebioglu, et al., 2018)
Carvacrol	---	(Zehra I. Yildiz et al., 2018)
Menthol	---	(Zehra Irem Yildiz et al., 2018)
Cineole	---	(Celebioglu, Yildiz, et al., 2018a)
p-cymene	---	(Celebioglu, Yildiz, et al., 2018a)
Eugenol	---	(Celebioglu, Yildiz, et al., 2018b)
Thymol	---	(Celebioglu, Yildiz, et al., 2018c)
Cinnamaldehyde	---	(Zehra I. Yildiz et al., 2019)
Living bacteria	Water – skim milk	(San Keskin et al., 2018)

	Acyclovir	---	(Celebioglu & Uyar, 2021)
	Kanamycin	---	(Topuz et al., 2021)
	Chloramphenicol	---	(Topuz et al., 2021)
	Gentamicin	---	(Topuz et al., 2021)
	Ampicillin	---	(Topuz et al., 2021)
SEB β CDs	Sulfisoxazole	---	(Zehra Irem Yildiz et al., 2017)
	Paracetamol	---	(Zehra Irem Yildiz & Uyar, 2019)
	Voriconazole	---	(Vass et al., 2019)
HP γ CDs	α -lipoic acid	---	(Celebioglu & Uyar, 2019a)
	Curcumin	---	(Celebioglu & Uyar, 2020c)
	Ferulic acid	---	(Celebioglu & Uyar, 2020e)
	Vanillin	Water, DMF, DMAc	(Celebioglu, Yildiz, et al., 2018b)
	Limonene	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Kusu, et al., 2016)
	Linalool	---	(Aytac, Yildiz, et al., 2017)
	Citral	---	(Aytac, Celebioglu, et al., 2018)
	Carvacrol	---	(Zehra I. Yildiz et al., 2018)
	Menthol	---	(Zehra Irem Yildiz et al., 2018)
	Cineole	---	(Celebioglu, Yildiz, et al., 2018a)
	p-cymene	---	(Celebioglu, Yildiz, et al., 2018a)
	Eugenol	---	(Celebioglu, Yildiz, et al., 2018b)
	Thymol	---	(Celebioglu, Yildiz, et al., 2018c)
	Cinnamaldehyde	---	(Zehra I. Yildiz et al., 2019)
M β CDs	Vitamin-A acetate	---	(Celebioglu & Uyar, 2020a)
	Vanillin	Water, DMF, DMAc	(Celebioglu, Kayaci-Senirmak, et al., 2016)
	Limonene	---	(Aytac, Yildiz, Kayaci-Senirmak, San Keskin,

				Kusku, et al., 2016)
		Linalool	---	(Aytac, Yildiz, et al., 2017)
		Citral	---	(Aytac, Celebioglu, et al., 2018)
		Eugenol	---	(Celebioglu, Yildiz, et al., 2018b)
		Thymol	---	(Celebioglu, Yildiz, et al., 2018c)
Air filter materials	γ CDs	---	Water – DMSO	(Celebioglu & Uyar, 2013b)
	HP β CDs	---	Water – DMF	(Celebioglu, Sen, et al., 2016)
	HP γ CDs	---	Water – DMF	(Celebioglu, Sen, et al., 2016)
Reducing agents for nanoparticle synthesis		Au-NPs	Water – DMF	(Celebioglu & Uyar, 2013c)
	HP β CDs	Ag-NPs	Water – DMF	(Celebioglu, Topuz, Yildiz, et al., 2019b)
		Pd-NPs	Water – DMF	(Celebioglu, Topuz, & Uyar, 2019a)
Sacrificial templates for ceramic hollow nanofibers	HP β CDs	---	DMF	(Celebioglu, Vempati, et al., 2014)
	M β CDs	---	DMF	(Celebioglu, Vempati, et al., 2014)
Water purification		---	---	(Celebioglu et al., 2017)
	PolyHP β CDs	Poly-benzoxazine	DFM	(Dogan et al., 2019)
		---	---	(Celebioglu, Topuz, Yildiz, et al., 2019a)
		---	Water – NaOH	(Celebioglu, Topuz, & Uyar, 2019b)
	Peracetylated β CDs	---	DMF	(Topuz et al., 2020)
Implantable scaffolds	PolyHP β CDs	Triclosan	---	(Hamsici et al., 2017)
		Azobenzene	Water – NaOH	(Heydari et al., 2018)

181 **4.1 Fundamental Studies**

182 The first report concerning polymer-free cyclodextrin electrospun nanofibers can be attributed to the group
183 of Uyar ten years ago, which investigated the electrospinning of methyl- β -cyclodextrins using water or
184 dimethylformamide (DMF) as solvents with a concentration ranging from 50 wt% to 61 wt% (i.e., 100-160
185 w/v%) (Celebioglu & Uyar, 2010). A few years later, the same authors extended their studies to three other
186 CD derivatives (i.e., HP β CDs, HP γ CDs and M β CDs) in different solvents (Celebioglu & Uyar, 2012).
187 Similarly to polymer-based systems, the results revealed that CD electrospinning is mainly governed by the
188 solution viscosity, bound water, and conductivity. Comparable findings were also reported by Manasco et al.
189 (Manasco et al., 2012), who investigated the possibility to prepare HP β CD-based nanofibers (Figure 4a-g) by
190 using aqueous solutions with a concentration in the range of 10-70 wt%.



191

192 **Figure 4.** (a) Effect of HP β CD concentration on the conductivity, bound water and viscosity of aqueous
193 solutions. SEM micrographs of electrospun HP β CD solutions of (b) 50 wt%, (c) 60 wt%, (d) 65 wt%, (e) and
194 70 wt%. TEM micrographs showing fibers electrospun from a 65 wt% solution at (f) 200000X and (g)
195 400000X magnifications. Reproduced with permission from Manasco et al., 2012. © The Royal Society of
196 Chemistry 2012.

197
198 In another work, Zhang et al. (W. Zhang et al., 2012) evaluated the influence of HP β CD concentration on the
199 electrospinning process in DMF. By studying the solution viscosity concentration dependence and the
200 concentration regimes, the authors supposed that HP β CDs behave in DMF as neutral polymers in good
201 solvents nevertheless much higher scaling factors were observed compared to the predicted ones (Colby,
202 2010). Such results were explained by considering the HP β CD tendency to self-aggregate similarly to
203 wormlike micelles (Cashion et al., 2010; Dodero, Vicini, Alloisio, et al., 2020; Rubinstein & Semenov,
204 2001). Celebioglu et al. (Celebioglu & Uyar, 2013a) also reported on the electrospinning of native α CDs and
205 β CDs from aqueous solutions in the concentration range 55-61 wt% (i.e., 120-160 w/v%). As for their
206 derivatives, the concentration of native cyclodextrin solutions was found to play a fundamental role in
207 conditioning the nanofiber morphology. Interestingly, besides the fact that native CDs are highly crystalline,
208 the authors proved that the resultant CD nanofibers were completely amorphous probably due to the fast
209 solvent evaporation rate. Yet, Ahn et al. (Ahn et al., 2013) presented the preparation of native β CD
210 nanofibers by using an ionic liquid and DMF as solvents and a concentration between 10 wt% and 60 wt%.
211 Only recently, Celebioglu et al. (Celebioglu & Uyar, 2020b) investigated the possibility to prepare
212 nanofibrous membranes by using highly concentrated (i.e., 61-71 wt% or 160-240 w/v%) hydroxypropyl-
213 α CDs (HP α CDs) solutions. Noticeably, the authors also considered industrially relevant aspects by using
214 distilled water, tap water and salty water for preparing the nanofibrous membranes. Furthermore, the effect
215 on the electrospinning process of solution storage over 8 days was investigated due to the tendency of CDs to
216 form aggregates, despite no significant changes were observed neither in the viscosity of the solutions nor in
217 terms of electrospun fiber morphology.

218

219 **4.2 Applicative Studies**

220 The previously discussed pioneering works demonstrated the possibility to fabricate polymer-free
221 cyclodextrin nanofibers straightforwardly by using different types of CDs and solvents, thereby anticipating
222 the development of a broad number of applicative products.

223

224 4.2.1 Drug and Bioactive Compound Delivery Systems with Enhanced Release Performances

225 The ability of CDs to increase the water solubility of several hydrophobic molecules, as well as their thermal
226 resistance, light stability, and bioavailability, has led to the development of polymer-free nanofibers with
227 enhanced drug delivery capabilities hence allowing the development of a new generation of biomedical and
228 pharmaceutical devices.

230 Encapsulation of Hydrophobic Drugs

231 Celebioglu et al. (Celebioglu, Umu, et al., 2014; Celebioglu & Uyar, 2011) first prepared HP β CD-ICs-
232 triclosan and HP γ CD-ICs-triclosan (TCL) electrospun membranes via the electrospinning of polymer-free
233 aqueous solutions achieving defect-free homogeneous nanofibers, with similar morphological results
234 obtained with poly(lactic acid)-based nanofibers in the work of Kayaci et al. (Kayaci, Umu, et al., 2013). The
235 authors investigated the CD-ICs-TCL formation to select the optimal compound concentration to obtain
236 homogeneous nanofibers while avoiding the presence of free triclosan molecules. Both HP β CD-ICs-TCL
237 and HP γ CD-ICs-TCL nanofibers were tested against E. Coli and S. Aureus bacteria showing enhanced and
238 prolonged antibacterial properties compared to uncomplexed triclosan powder. Remarkably, Chen et al. (M.
239 Chen et al., 2013) reported on the preparation of UV-responsive nanofibers consisting of the inclusion
240 complexes between HP β CDs and azobenzene (AAB) molecules. By investigating the effect of UV light in
241 different phases of the electrospinning process, the authors were able to prove the responsive properties of
242 the prepared mats which were pointed out to be extremely promising for the development of stimuli-
243 controlled drug delivery systems.

245 Encapsulation of Volatile Bioactive Substances

246 Polymer-free cyclodextrin nanofibers can also be efficiently exploited to avoid the rapid evaporation of
247 highly volatile compounds. For example, Aytac et al. (Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Tekinay,
248 et al., 2016) prepared HP β CD, HP γ CD, or M β CD geraniol inclusion complex nanofibrous mats. The authors
249 proved the capability to encapsulate a higher amount of geraniol in pure CD nanofibers (~11 wt%) compared
250 to polymeric membranes (~5 wt%). After both short-term temperature and long-term open-air release studies,

251 M β CD-based nanofibers showed the slowest release of geraniol. Moreover, along with strong antibacterial
252 activities, CD-ICs-geraniol mats also displayed efficient antioxidant properties. Yet, Celebioglu et al.
253 (Celebioglu, Aytac, et al., 2018) prepared HP β CD-ICs- and HP γ CD-ICs-camphor nanofibers from highly
254 concentrated aqueous solutions obtaining thermally stable and manageable membranes, with HP γ CD-ICs-
255 camphor nanofibers showing a slow release of the compound.

256

257 4.2.2 *Fast-dissolving Nanofibers for the Rapid Compound Administration in Food or Pharmaceutical* 258 *Products*

259 Polymer-free cyclodextrin nanofibers are highly soluble in water if not subjected to a crosslinking reaction
260 (see Section 5), but such drawback has been efficiently turned into a benefit by exploiting the fast-dissolving
261 nature of these systems for the rapid administration of drugs and bioactive compounds in both food and
262 pharmaceutical products (Balusamy et al., 2020). Owing to their capability to form stable inclusion
263 complexes, cyclodextrins can indeed considerably increase the water-solubility of a wide variety of guest
264 hydrophobic drugs and volatile molecules that can consequently be rapidly released when in contact with a
265 high moisture environment, human saliva or biological fluids (Cheirsilp & Rakmai, 2017; Mura, 2015).

266

267 *Encapsulation for the Fast-dissolution of Hydrophobic Substances*

268 The encapsulation of a hydrophobic drug within polymer-free cyclodextrin membranes has been first
269 demonstrated by Vigh et al. (Vigh et al., 2013). The authors reported on polymer-free HP β CD nanofibers
270 encapsulating spironolactone which were able to completely release the encapsulated drug within a minute,
271 therefore being promising for the rapid oral administration of highly lipophilic compounds. Shortly after,
272 Balogh et al. (Balogh et al., 2015) investigated the possibility to prepare fast-dissolving HP β CD nanofibers
273 containing diclofenac sodium (DCF), a poorly soluble and highly crystalline drug, to avoid the common
274 issues related to its injection delivery methods. Remarkably, the authors implemented the common
275 electrospinning set-up with high-speed blowing air (i.e., electroblowing) able to enhance both productivity
276 rate and fiber morphology. Above all, HP β CD-ICs-DCF nanofibers were completely amorphous and
277 displayed a high solubility in water hence representing a promising alternative to reconstitution injections.

278 Starting from these preliminary studies, great efforts have been done in preparing fast-dissolving
279 cyclodextrin electrospun nanofibers encapsulating a variety of hydrophobic molecules. For instance,
280 Celebioglu et al. (Celebioglu & Uyar, 2017) reported on the electrospinning of HP β CD inclusion complexes
281 with vitamin E, which consists of a long hydrophobic chain. The authors were able to load and preserve
282 around the 13 wt% of vitamin E in the prepared mats observing only a small loss of the compound during the
283 electrospinning process. The mats showed a fast-dissolving nature and strong antioxidant activities, which
284 were even maintained after UV-irradiation and 3-year storage proving the stability of the formed complexes.
285 Relatedly, Yildiz et al. (Zehra Irem Yildiz et al., 2017) developed first a nanofibrous solid inclusion complex
286 between sulfobutylether- β -cyclodextrins (SEB β CDs) and sulfisoxazole, and then HP β CD-ICs- and
287 SBE β CD-ICs-paracetamol nanofibrous membranes (Zehra Irem Yildiz & Uyar, 2019). The authors
288 demonstrated that the crystalline nature of paracetamol was completely lost owing to the complexation with
289 CDs, thus increasing its water solubility, bioavailability and thermal stability. Recently, Celebioglu et al.
290 (Celebioglu & Uyar, 2019a, 2019b, 2019c, 2020c, 2020d, 2020e) reported on several types of polymer-free
291 cyclodextrin nanofibers encapsulating hydrophobic drugs showing a fast-dissolving nature. For example, the
292 authors demonstrated the possibility to form stable HP β CD-ICs- and HP γ CD-ICs- α -lipoic acid nanofibrous
293 membranes. Owing to the absence of crystallinity, the prepared mats displayed a fast-dissolving nature both
294 in water and artificial saliva, and they were characterized by good mechanical resistance. Similarly, the
295 authors prepared free-standing and flexible HP β CD-ICs-ibuprofen nanofibers achieving the complete
296 inclusion of the drug within the host molecules and a much higher loading concentration compared to
297 PVA/CD-ICs-ibuprofen nanofibers investigated in the same work. Again, metronidazole, a poorly water-
298 soluble drug widely used in pharmaceutical formulations, was encapsulated in polymer-free HP β CD
299 nanofibers via the electrospinning of highly concentrated aqueous solutions. Lately, the authors also reported
300 on the preparation of nanofibrous membranes consisting of the inclusion complex between HP β CDs or
301 HP γ CDs and curcumin with a remarkable 100% encapsulation efficiency. Specifically, HP γ CD-ICs-
302 curcumin nanofibers displayed a much higher water solubility and were almost instantaneously dissolved
303 once in contact with water or artificial saliva. Moreover, the authors were able to prove the enhanced
304 antioxidant properties of the prepared mats compared to pristine curcumin, with similar results reported by

305 Aytac et al. (Aytac & Uyar, 2017). In an analogous study, the authors prepared electrospun HP β CD-ICs-
306 hydrocortisone nanofibers to enhance the water solubility and the bioavailability of the drug obtaining self-
307 standing and flexible defect-free mats able to completely preserve the drug during the electrospinning
308 process. Additionally, the authors investigated the preparation of nanofibrous mats consisting of the
309 inclusion complex between ferulic acid (FA) and HP β CDs or HP γ CDs. Despite both systems allowed to
310 reach a compound content of ~11 wt%, HP γ CD-ICs-ferulic acid were found to be much more stable thereby
311 making it possible to obtain amorphous nanofibers with enhanced thermal stability and fast-dissolving nature.
312 Lately, Gao et al. (Gao et al., 2020) prepared HP β CD-ICs-thiabendazole (TBZ) nanofibrous mats to exploit
313 the compound antifungal properties, which are usually limited by its poor solubility. Interestingly, owing to
314 the fast-dissolving nature of the mats, the antifungal properties of the compound were considerably enhanced
315 thereby indicating the possibility to use such systems as a novel pesticide formulation. Yet, Celebioglu et al.
316 (Celebioglu & Uyar, 2020a) demonstrated the possibility to prepare HP β CD-ICs- and HP γ CD-ICs-vitamin A
317 acetate nanofibers with self-standing and fast-dissolving nature. In detail, the nanofibers were prepared with
318 a loading concentration of around 5 wt% with the complexation between the HPCDs and the bioactive
319 compound ensuring the fast dissolution both in water and artificial saliva media. Only recently, Celebioglu et
320 al. (Celebioglu & Uyar, 2021) also reported on polymer-free HP β CD-ICs-acyclovir nanofibers. In particular,
321 by using PVP-acyclovir nanofibrous mats as a control sample, the authors proved the greater encapsulation
322 efficiency of the hydrophobic compound within the membranes in the presence of the inclusion complexes.
323 Additionally, Topuz et al. (Topuz et al., 2021) also developed polymer-free HP β CD nanofibers with high
324 antibiotic loadings through the electrospinning of cyclodextrin-antibiotic inclusion complexes from water,
325 and the resultant fast-dissolving fibers demonstrated high antibacterial activity towards Gram-negative
326 bacteria.

327 Remarkably, the possibility to prepare polymer-free cyclodextrin nanofibers at an industrial scale has also
328 been explored, despite only a few works are reported in the literature. In this regard, Vass et al. (Vass et al.,
329 2019) investigated the use of a high-speed electrospinning (HSES) setup to process an aqueous solution of
330 SBE β CD-ICs-voriconazole (VOR). The authors were able to achieve an outstanding production rate of 300
331 mL/h (i.e., 240 g/h of solid material) under ambient conditions and the grounded fibrous powder showed to

332 be rapidly dissolvable in water with similar results to Vfend[®] (i.e., reference commercial pharmaceutical
333 product containing VOR), consequently demonstrating the capability of the proposed approach to fulfil the
334 requirements of the pharmaceutical industry. More recently, the authors also reported on the high-speed
335 electrospinning of HP β CD nanofibers containing β -galactosidase, which is a protein-type drug widely used
336 in pharmaceutical formulations, that were subsequently grounded and converted in tablets for oral
337 assumption with β -galactosidase found to maintain its activity over a period of 6 months (Vass et al., 2020).
338 Similarly, Kiss et al. (Kiss et al., 2020) developed a solid HP β CD-ICs-doxycycline (DOX) intravenous
339 formulation using high-speed electrospinning with a production rate of \sim 80 g/h (i.e., solid material). The
340 authors proved that the explored inclusion complex allowed the preparation of a DOX formulation with a
341 concentration 7 times higher than the commercially available ones, hence suggesting HSES as an alternative
342 to freeze-drying technique.

343

344 *Encapsulation for the Fast-dissolution of Volatile Substances*

345 Another important advantage of polymer-free cyclodextrin nanofibers is their ability to encapsulate highly
346 volatile compounds hence preserving their bioactivity over a long period while ensuring their fast
347 administration when required. For example, Celebioglu et al. (Celebioglu, Kayaci-Senirmak, et al., 2016)
348 prepared polymer-free electrospun mats by using the inclusion complexes between vanillin and HP β CDs,
349 HP γ CDs, and M β CDs in water, DMF or DMA. The authors demonstrated that M β CDs were more stably
350 linked to vanillin leading to a higher encapsulation efficiency (up to \sim 12 wt%). Remarkably, compared to
351 pure PVA and PVA/CD-ICs-vanillin nanofibers (Kayaci & Uyar, 2012b), the proposed CD-based mats could
352 encapsulate a considerably higher amount of vanillin and showed strong antioxidant activities, high thermal
353 stability and the possibility to tune their dissolving behaviour. Similarly, Aytac et al. (Aytac, Yildiz, et al.,
354 2017; Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Kusku, et al., 2016) proposed the preparation of
355 polymer-free HP β CD-, HP γ CD-, and M β CD-based nanofibrous mats encapsulating the highly volatile
356 limonene or linalool compounds. In both cases, the authors proved M β CD nanofibers to be the most efficient
357 in terms of encapsulation capability. Remarkably, short-term (i.e., 3h) and long-term (i.e., 100 days) release
358 studies demonstrated the stability of the encapsulated compounds within the nanofibrous mats, which

359 showed strong antibacterial activities against both Gram-positive and Gram-negative bacteria, as well as
360 good manageability and fast-dissolving character in water or high humidity environment. In another study,
361 the authors reported on the encapsulation of citral within HP β CD-, HP γ CD- and M β CD-based nanofibers
362 obtaining an 80% encapsulation efficiency and the capability to preserve half of the volatile compound after
363 15 days, as well as increasing both its thermal stability and water solubility (Aytac, Celebioglu, et al., 2018).
364 Comparably, fast-dissolving polymer-free HP β CD-ICs- and HP γ CD-ICs-carvacrol nanofibers with potential
365 applications as food or oral care products were reported by Yildiz et al. (Zehra I. Yildiz et al., 2018). Besides
366 the high encapsulation efficiency, the carvacrol complexation enhanced its thermal stability and antioxidant
367 properties for up to three years. In a similar study, the authors explored the preparation of polymer-free CD-
368 ICs-menthol nanofibers by using highly concentrated HP β CD-IC and HP γ CD-IC aqueous solutions (Zehra
369 Irem Yildiz et al., 2018). The stable complexation of menthol, a volatile substance with strong antibacterial
370 properties widely used in several applications, led to an increase of the compound solubility and thermal
371 stability while allowing to obtain homogenous and smooth nanofibers with good flexibility and a fast-
372 dissolving character. The developed mats endorsed to encapsulate a much higher amount of menthol, which
373 was also stable over a long period, compared to polystyrene (PS)/CD-ICs-menthol (Uyar, Hacaloglu, et al.,
374 2009) and polymethylmethacrylate (PMMA)/CD-ICs-menthol (Uyar, Nur, et al., 2009) nanofibers. Also,
375 Celebioglu et al. (Celebioglu, Yildiz, et al., 2018a) prepared HP β CD-ICs- and HP γ CD-ICs-based electrospun
376 mats containing cineole or p-cymene. HP γ CD-based nanofibers were able to encapsulate a greater amount of
377 cineole, whereas p-cymene was better preserved by the HP β CD-based ones. However, both mats improved
378 the compound thermal stability consequently representing potential products in food and oral care
379 applications. Yet, the authors investigated the preparation of free-standing nanofibrous mats obtained via the
380 electrospinning of HP β CD-ICs-, HP γ CD-ICs-, and M β CD-ICs-eugenol or thymol (THY) systems
381 (Celebioglu, Yildiz, et al., 2018b, 2018c). The complex formation within the nanofibers allowed to reach an
382 encapsulation efficiency considerably higher compared to analogous PVA/CDs-ICs-eugenol nanofibers
383 (Kayaci, Ertas, et al., 2013). Additionally, owing to their fast-dissolving nature, CD-ICs-eugenol and CD-
384 ICs-thymol nanofibers showed much stronger antioxidant properties compared to the pure compounds. In
385 another work, cinnamaldehyde was successfully encapsulated in self-standing HP β CD- and HP γ CD-based

386 nanofibers by Yildiz et al. (Zehra I. Yildiz et al., 2019). Bead-free and homogeneous nanofibers were
387 obtained with considerably higher efficiency in preserving the volatile cinnamaldehyde compared to
388 analogous PLA-based nanofibers (Y. Liu et al., 2017). Moreover, owing to their fast-dissolving nature, the
389 prepared mats displayed strong antibacterial properties against E. Coli representing a new potential class of
390 materials for applications in both the food, biomedical and pharmaceutical industries.

391

392 *Encapsulation of Living Bacteria*

393 Keskin et al. (San Keskin et al., 2018) demonstrated the possibility to encapsulate living bacteria within
394 electrospun HP β CD-based nanofibers to increase their availability for wastewater bioremediation
395 applications. Despite the encapsulation efficiency was not particularly high, a sufficient amount of bacteria
396 was preserved during the preparation process with the cell viability maintained for 7 days. Remarkably, the
397 used bacteria retained their bioactivity and were proved able to remove Ni(II), Cr(VI) and a commercial
398 reactive dye from aqueous solutions. Such findings indicated the proposed approach as a faster and simpler
399 alternative to the lyophilization procedure commonly applied to preserve organisms for strain collections and
400 applications in dye removal products.

401

402 *4.2.3 Applications in Air and Water Filtration*

403 The capability of cyclodextrins and their derivatives to form stable non-covalent inclusion complexes with a
404 variety of molecules makes them extremely interesting for the removal of several pollutants from the
405 environment. Furthermore, nanofibrous electrospun membranes represent a class of materials with superior
406 adsorption capabilities owing to their unique structure, which allows exploiting a higher surface of
407 interaction between the adsorbent and the adsorbate. With these premises, in the past decade, intensive
408 research has been carried out to develop efficient filter materials based on polymer-free cyclodextrin
409 nanofibers aiming to maximize the adsorption capability. Celebioglu et al. (Celebioglu & Uyar, 2013b) first
410 reported on the electrospinning of pristine γ CDs in dimethyl sulfoxide (DMSO) aqueous solutions to be used
411 for the entrapment of volatile organic compounds (VOCs). The obtained electrospun mats were completely
412 amorphous and presented, above all, a surface area three times higher than their powder form according to

413 BET analysis. Aniline and toluene volatile organic compounds were used to test the adsorption ability of
414 γ CD nanofibers that showed a marked complexation capability compared to γ CD powder. Similarly, the
415 authors also investigated the possibility to use HP β CD- and HP γ CD-based nanofibers as filter materials
416 (Celebioglu, Sen, et al., 2016). Polymer-free electrospinning of CDs was carried out in water and DMF
417 obtaining defect-free mats with a considerably higher surface area with respect to CD powders. DMA
418 characterization was employed to demonstrate the good mechanical properties and integrity of the obtained
419 mats, which were indicated as potentially foldable materials to be inserted in air filter modules. Recently,
420 Topuz et al. (Topuz et al., 2020) reported on hydrophobic water-insoluble nanofibers obtained from the
421 polymer-free electrospinning of peracetylated β -cyclodextrins. Native β CDs were first modified and
422 subsequently electrospun by using a DMF solution, whose minimum concentration to obtain defect-free
423 nanofibers was ~64 wt% (i.e., 180 w/v). The peracetylation procedure drastically boosted the thermal
424 stability and the hydrophobicity of the obtained nanofibers, which were indeed being proved to maintain
425 their nanofibrous structure in water for 24h.

426

427 4.2.4 Other Applications

428 Despite the capability to form inclusion complexes that remains one of the most interesting and exploited
429 properties of cyclodextrins, other promising applications have been considered as well. For example, their
430 use for the easy synthesis of metal nanoparticles have been also investigated (He et al., 2019; Prochowicz et
431 al., 2016, 2017). CDs can indeed directly act as reducing agents for metal ions without the need for toxic
432 chemicals or severe processing conditions, hence allowing the development of environmentally benign
433 materials with unique features and a wide range of possible applications (Celebioglu, Aytac, et al., 2014;
434 Shan Wang, Bai, Li, & Zhang, 2012; Shan Wang, Bai, Li, Zhang, et al., 2012). Celebioglu et al. (Celebioglu
435 & Uyar, 2013c) first proposed the one-step in-situ synthesis of gold nanoparticles (Au-NPs) via the
436 electrospinning of HP β CD solutions containing an appropriate salt precursor. The authors exploited the
437 capability of CDs to act as reducing and stabilizing agents, as well as fibrous matrix supporting material, to
438 synthesize Au-NPs directly during the electrospinning process. Subsequently, the authors reported on the
439 preparation of HP β CD nanofibers containing in-situ synthesized silver nanoparticles (Ag-NPs) produced by

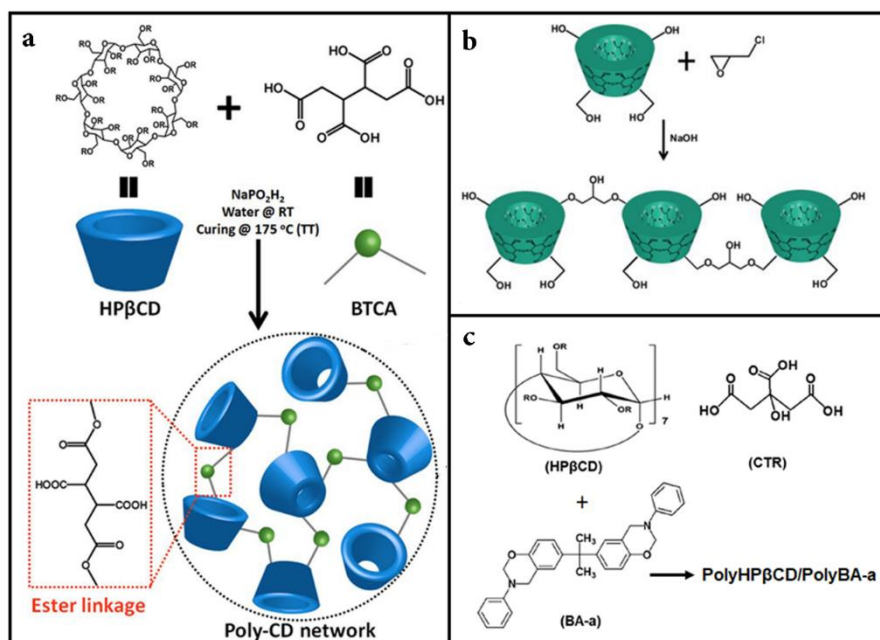
440 a single-step electrospinning process (Celebioglu, Topuz, Yildiz, et al., 2019b). The authors observed that
441 DMF solution led to thinner nanofibers compared to water, besides an increase of the mean diameter was
442 observed for both solvents by increasing the amount of loaded Ag salt precursor, as similarly reported by
443 Chae et al. (Chae et al., 2011). TEM images revealed that Ag-NPs having a size between 2 and 5 nm were
444 successfully embedded within the nanofibers, with their dimension being a function of the precursor amount
445 and solvent type. Owing to the nanoparticle presence, the prepared nanofibers showed strong antibacterial
446 properties against *E. Coli* and *S. Aureus* bacteria over a period of 24h, thus being suitable also for biomedical
447 and food packaging applications. Yet, the authors proposed HP β CD nanofibers loaded with palladium
448 nanoparticles (Pd-NPs) (Celebioglu, Topuz, & Uyar, 2019a). Cyclodextrin solutions in water and DMF
449 containing an appropriate precursor were electrospun leading to bead-free nanofibers. Pd-NPs were
450 homogeneously distributed within the nanofibers and showed a dimension ranging from 3 up to 5 nm that
451 increased with the precursor loading concentration and was bigger for DMF compared to water. Moreover,
452 HP β CD/Pd-NP nanofibers showed strong catalytic performances being able to reduce p-nitrophenol (PNP)
453 in few minutes into p-aminophenol (PAP), thus showing great potentialities for the development of products
454 for catalytic applications.

455 Cyclodextrin-based nanofibers, owing to their fast-dissolving character in water, can be even employed as
456 sacrificial templates for the development of hollow nanofibers. To this purpose, Celebioglu et al. (Celebioglu,
457 Vempati, et al., 2014) reported on polymer-free cyclodextrin nanofibers (i.e., HP β CDs and M β CDs) as a
458 sacrificial pattern for the preparation of metal oxide nanotubes via atomic layer deposition (ALD). The
459 authors proved the possibility to deposit a layer of aluminium oxide (Al₂O₃) or zinc oxide (ZnO) on the
460 nanofiber surface to obtain a core-shell structure, subsequently removing the cyclodextrin core by a simple
461 washing process in water. The presence of -OH groups was found to promote the smooth and homogeneous
462 deposition as shown by SEM images, whereas TEM analysis indicated that Al₂O₃ and ZnO maintained an
463 amorphous and highly crystalline nature with wurtzite structure. Despite such an approach remains at a
464 preliminary stage, the obtained findings indicated that the use of water-soluble CD nanofibers could lead to
465 the simple and cost-effective preparation of metal oxide nanotubes with possible energy storage applications
466 (Yun et al., 2020).

467

468 **5 Water-insoluble Crosslinked Polycyclodextrin Nanofibers**

469 The high surface area of cyclodextrin-based electrospun mats and their unique inclusion complexation
470 capability make such systems very promising for wastewater purification applications, as well as for
471 implantable biomedical devices. However, as broadly discussed in the previous Section, their high solubility
472 and fast-dissolving features in aqueous environments represent a limitation to their proficient use for such
473 applications. Therefore, the development of crosslinked cyclodextrin-based systems has started to be
474 explored in the viewpoint to fabricate insoluble and highly performing systems. However, as summarized in
475 Table 1, only a few studies are reported in the literature and further research are needed to investigate
476 eventual toxicity issues related to the crosslinking reaction. Figure 5 depicts some of the most commonly
477 employed crosslinking approaches for cyclodextrins.



478

479 **Figure 5.** Schematics of different crosslinking strategies of the cyclodextrin nanofibers. The crosslinking
480 agents are added to the CD solution prior to electrospinning and a curing step initiate the reaction in the final
481 membrane. Reaction of CDs with (a) 1,2,3,4-butanetetracarboxylic acid (BTCA) (reproduced with
482 permission from Celebioglu et al., 2017. © The Author(s) 2017), (b) epichlorohydrin (ECH) (reproduced
483 with permission from Celebioglu, Topuz, & Uyar, 2019b. © 2018 American Chemical Society), and (c) citric
484 acid (CTR) and benzoxazine (BA-a) monomers (reproduced with permission from Dogan et al., 2019. ©
485 2019 Elsevier Ltd).

486

487 **5.1 Water Purification Applications of polyCD Nanofibers**

488 Celebioglu et al. (Celebioglu et al., 2017) first demonstrated the possibility to prepare crosslinked
489 electrospun poly-cyclodextrin nanofibers as highly efficient molecular filter materials. By using

490 butanetetracarboxylic acid (BTCA) as crosslinker (Figure 5a), the authors avoided the fast dissolution of the
491 prepared polyHP β CD nanofibers while maintaining their high hydrophilicity. In particular, polyHP β CD
492 nanofibers were characterized by a good affinity with methylene blue (MB) having an extremely high
493 adsorption capacity at dye concentration well above the acceptable limits, showing improved performances
494 compared to an acetate cellulose commercial filter and similar polyacrylonitrile (PAN)/CD nanofibers (W.
495 Zhang et al., 2019). Moreover, the authors were able to prove both their reusability via a simple, fast, and
496 cost-effective washing cycle and their applicability in a real water filtration apparatus. Similarly, Dogan et al.
497 (Dogan et al., 2019) reported on polyHP β CD/poly-benzoxazine (polyBA-a) nanofibers as potential dye
498 adsorbents by using citric acid (CTR) as crosslinker and activating the crosslinking reaction via a simple
499 thermal treatment (Figure 5c). The authors proved that the crosslinked mats were able to withstand several
500 washing cycles with both water and organic solvents showing enhanced thermal stability, hence representing
501 promising products for filtration applications. Noticeably, the obtained mats were tested against a mixture of
502 methylene blue and methyl orange (MO) dyes exhibiting a strong selectivity towards MB being able to
503 nearly completely remove the dye in a period of 6h. Yet, Celebioglu et al. (Celebioglu, Topuz, Yildiz, et al.,
504 2019a) evaluated the possibility to use the polyHP β CD nanofibers crosslinked with BTCA as filter materials
505 for the removal of several polycyclic aromatic hydrocarbons (PHAs) and heavy metals from water obtaining
506 interesting equilibrium adsorption capabilities and the ability to reduce to half the contaminant amount in
507 only 20 minutes. Interestingly, the proposed mats could be recycled four times by a simple treatment with
508 acetonitrile or with a nitric acid solution without showing changes in their morphology and adsorption
509 capability. Also, the authors recently reported on water-insoluble polyHP β CD nanofibers obtained by using
510 epichlorohydrin (ECH) as crosslinker agent (Figure 5b) (Celebioglu, Topuz, & Uyar, 2019b). The
511 rheological investigation of HP β CD/ECH aqueous solutions showed that the reaction partially initiated prior
512 to the electrospinning, despite a post-preparation thermal treatment was the main responsible for the process.
513 The obtained polyHP β CD nanofibers were proved to be highly hydrophilic, nearly completely amorphous,
514 with enhanced thermal stability, and were above all able to maintain their structure after the immersion in
515 water and several organic solvents. The authors investigated the scavenging ability of the crosslinked
516 nanofibers against phenolphthalein and phenanthrene in their aqueous solutions proving that the prepared

517 nanofibers showed enhanced adsorption capabilities compared to polyHP β CD powder and analogous
518 poly(ethylene terephthalate) (PET)/CD nanofibers (Kayaci, Aytac, et al., 2013).

519

520 **5.2 Biomedical Applications of polyCD Nanofibers**

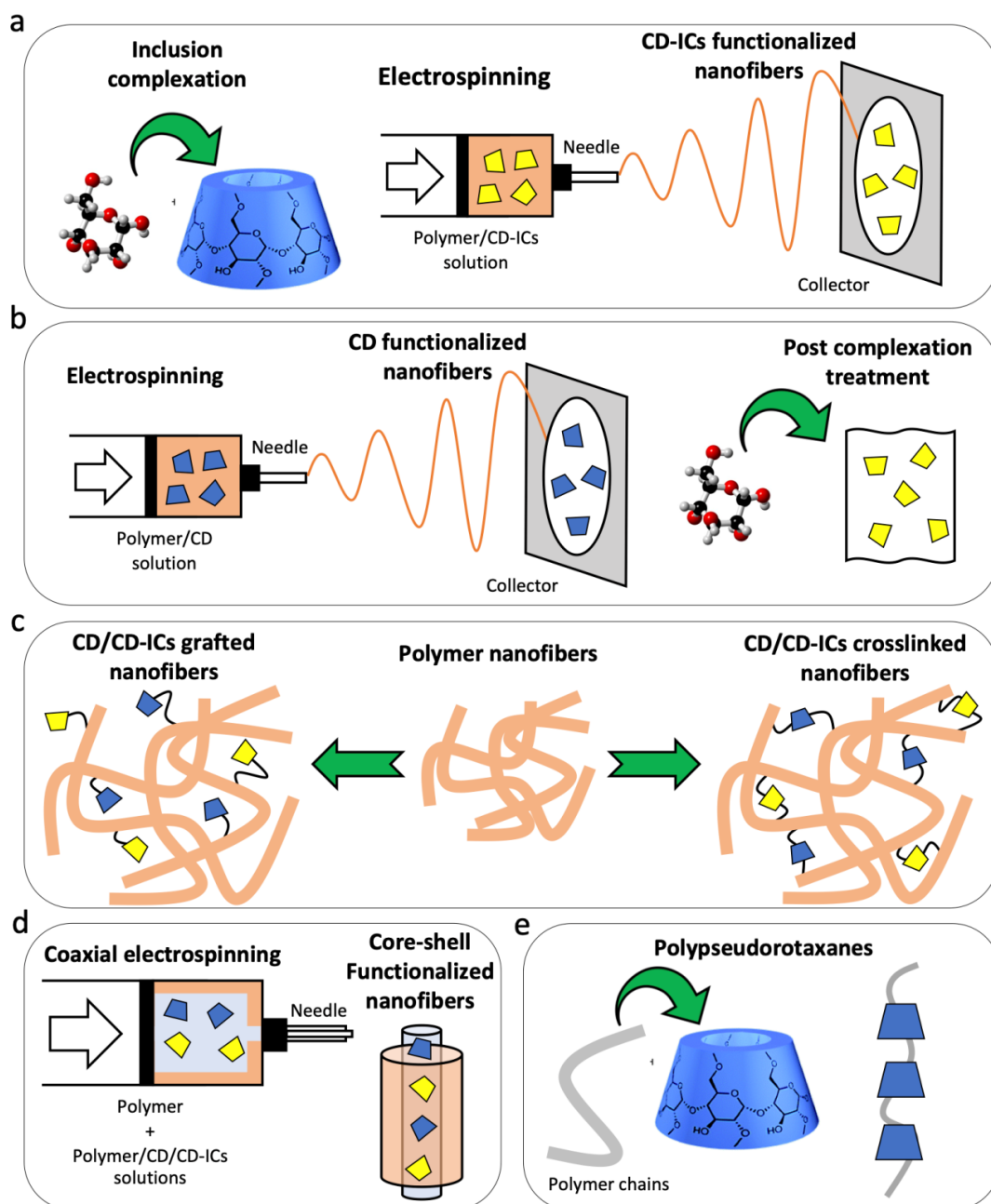
521 Hamsici et al. (Hamsici et al., 2017) proposed water-insoluble HP β CD aligned nanofibers functionalized
522 with bioactive peptides as implantable scaffolds for peripheral nerve regeneration. The highly aligned
523 nanofibers were first prepared and crosslinked via BTCA before being functionalized with IKVAV or KK
524 peptides with a simple impregnation procedure. PolyHP β CD-ICs-peptide nanofibers did not show toxicity
525 issues and provided the optimal environment to promote cell adhesion, differentiation and proliferation.
526 Specifically, the peptide presence on the nanofiber surface was found to guide the differentiation to PC-12
527 cells compared to unfunctionalized membranes, whereas the aligned topological arrangement was proven to
528 help the formation of longer neurites compared to the randomly structured samples. In another work, Heydari
529 et al. (Heydari et al., 2018) prepared hydrophobic peracetyl- β CD nanofibers loaded with vitamin B₂ (VB₂)
530 by the electrospinning of an acetone/DMF solution. HP β CDs were subjected to crosslinking and
531 peracetylation reactions before being electrospun together with VB₂ leading to bead-free homogeneous
532 nanofibers. PolyHP β CD-ICs-VB₂ nanofiber release kinetic was studied in simulated gastric and intestinal
533 conditions showing an initial burst release followed by a sustained slow release for 16h. Compared to
534 HP β CD-ICs-VB₂ powder, the prepared mats displayed a considerably slower and more controlled release of
535 vitamin B₂, thus representing an important advancement in the development of a new class of controlled drug
536 delivery systems.

537

538 **6 Electrospun Membranes Made of Natural Polymers Functionalized with Cyclodextrins and Their** 539 **Inclusion Complexes**

540 Together with polymer-free CDs and polyCDs nanofibers, electrospun mats containing cyclodextrins have
541 also been prepared from both native CDs and their derivatives in combination with a broad variety of carrier
542 polymers able to provide a good spinnability and very specific properties of the mats depending on the
543 targeted application (Costoya et al., 2017; Topuz & Uyar, 2019). The use of CDs to functionalize nanofibers

544 offers a wide number of advantages and represents a more and more investigated research field owing to the
545 wide range of possibilities of the obtained products. The ability of CDs to complex hydrophobic compounds
546 and volatile molecules represents the opportunity to enhance their encapsulation efficiency, stability,
547 solubility and bioavailability, as well as provide them with controlled release kinetics. For example, such
548 materials can be used as implantable scaffolds enriched with growth factors and vitamins, pharmaceutical
549 formulations containing poorly soluble drugs, highly performing filters for wastewater and volatile organic
550 compounds, and active food packaging materials with antibacterial and antioxidant properties (Alvarez-
551 Lorenzo et al., 2017; Celebioglu, Sen, et al., 2016; Hall Barrientos et al., 2017; Lin et al., 2017; Moyers-
552 Montoya et al., 2016; Wen, Zhu, Feng, et al., 2016). Several approaches are reported in the literature to
553 prepare cyclodextrin-functionalized electrospun nanofibers with some of them schematically illustrated in
554 Figure 6. The simplest one consists of adding to a proper polymer solution the CDs or the CD-ICs of interest,
555 with the latter usually prepared by precipitation or freeze-drying methods (Beachley & Wen, 2010; Meinel et
556 al., 2012). A faster alternative, which is however less applied owing to the difficulty in accurately control the
557 guest molecule loading concentration, consists of the direct mixing of all the components (i.e., polymer,
558 cyclodextrins, drug or bioactive compound, etc.) (Narayanan et al., 2017). Another possibility is represented
559 by the electrospinning of a polymer-cyclodextrins mixture with the a posteriori loading of the guest
560 molecules of interest usually via a solution impregnation method (Séon-Lutz et al., 2019). Yet, polymer
561 nanofibers can be functionalized by grafting their surface with CDs or CD-ICs via a chemical reaction,
562 therefore obtaining a high surface reactivity (Wu et al., 2015; Yuan et al., 2017). Similarly, CDs and their
563 derivatives can be directly employed as a crosslinker for the polymer chains, thus acting as stabilizing agents
564 and reducing the nanofiber solubility (Boas et al., 2015; L. Li & Hsieh, 2005). Alternatively, CDs and their
565 complexes can be completely embedded within the nanofibers by employing a coaxial set-up producing
566 core-shell nanofibers (Chou et al., 2015). Finally, it should be kept in mind that CDs and CD-ICs, when
567 mixed with a polymer in a liquid solvent, can thread along the polymer chains forming polypseudorotaxanes.
568 These can assemble in supramolecular structures where the interactions between CDs play a fundamental
569 role in conditioning the spinning solution properties (Uyar et al., 2008) and bring grafting sites for nanofiber
570 post-functionalization (M. Oster et al., 2017; Murielle Oster et al., 2015).



571

572 **Figure 6.** Schematics of preparation methods for cyclodextrin-functionalized nanofibers. (a) Electrospinning of polymer/CD-ICs solutions, (b) electrospinning of polymer/CDs/active compound solutions, (c) CD/CD-ICs grafted or CD/CD-ICs crosslinked nanofibers, (d) coaxial electrospinning of CD/CD-ICs core functionalized nanofibers, and (e) pseudopolyrotaxanes structures.

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To date, both synthetic and natural polymer-based nanofibers have been functionalized with cyclodextrins and their derivatives with their advantages and disadvantages. Along with the fact that most natural polymers can be processed in green solvents under mild working conditions, their main advantages are enhanced biocompatibility, biodegradability and the possibility to be obtained from agricultural and food wastes with the development of green and circular economy products. Thereby, it is much likely that natural polymer-

582 based electrospun membranes functionalized with cyclodextrins will soon represent a new class of
583 environmentally friendly materials with potential applicability in a wide range of markets. In the next two
584 subsections, the current literature about polysaccharide- and protein-based functionalized electrospun
585 membranes is critically discussed by highlighting the recent advances.

586

587 **6.1 Electrospun Polysaccharide – Cyclodextrin Based Membranes**

588 Polysaccharides are a family of natural polymers consisting of multiple saccharide units linked together
589 through glycosidic linkages and possess a wide variety of unique properties which make them extremely
590 interesting from an industrial point of view (Cazón et al., 2017; Dodero, Lova, Vicini, Castellano, et al.,
591 2020; Y. Yu et al., 2018). Most polysaccharides can be easily and cheaply recovered from natural sources
592 such as plants, microbes, and animals, with their use being especially profitable when they are by-products of
593 other processes (Dodero, Vicini, & Castellano, 2020; Hamed et al., 2016; Sila et al., 2014). Compared to
594 other natural polymers, polysaccharides are usually more stable and not irreversibly denatured upon heating
595 and can consequently be used as an effective shell to confer enhanced temperature stability to a variety of
596 bioactive compounds. Moreover, polysaccharides show a broad range of interesting properties such as
597 biocompatibility, low toxicity, mucoadhesive behaviour, biodegradability, and ease of functionalization,
598 hence being nowadays widely employed in several industrial fields (Öner et al., 2016). Regardless of their
599 advantages, the electrospinning of polysaccharides remains a challenge due to their tendency to form strong
600 hydrogen bonds that lead to high viscous solutions or even gels, the small number of entanglements formed
601 between the polymer chains, the high solution surface tension, and the poor water stability of the obtained
602 mats (Dodero, Vicini, et al., 2019; Mendes et al., 2017). For example, Dodero et al. reported on the
603 preparation of sodium alginate (SA) (Dodero, Alloisio, Vicini, & Castellano, 2020; Dodero, Donati, et al.,
604 2021; Dodero, Scarfi, Pozzolini, Vicini, et al., 2020) and chitosan (CH) (Dodero, Brunengo, Alloisio,
605 Sionkowska, et al., 2020; Dodero, Scarfi, et al., 2021) crosslinked electrospun membranes by using
606 poly(ethylene oxide) (PEO) as a co-spinning agent, which was then removed by a simple post-
607 electrospinning washing cycle. In another work, Séon-Lutz et al. (Séon-Lutz et al., 2019) proposed the
608 electrospinning of hyaluronic acid (HA)/PVA solutions being able to decrease the co-spinning agent
609 concentration compared to other similar works reported in the literature (J. Li et al., 2006), whereas Vicini et

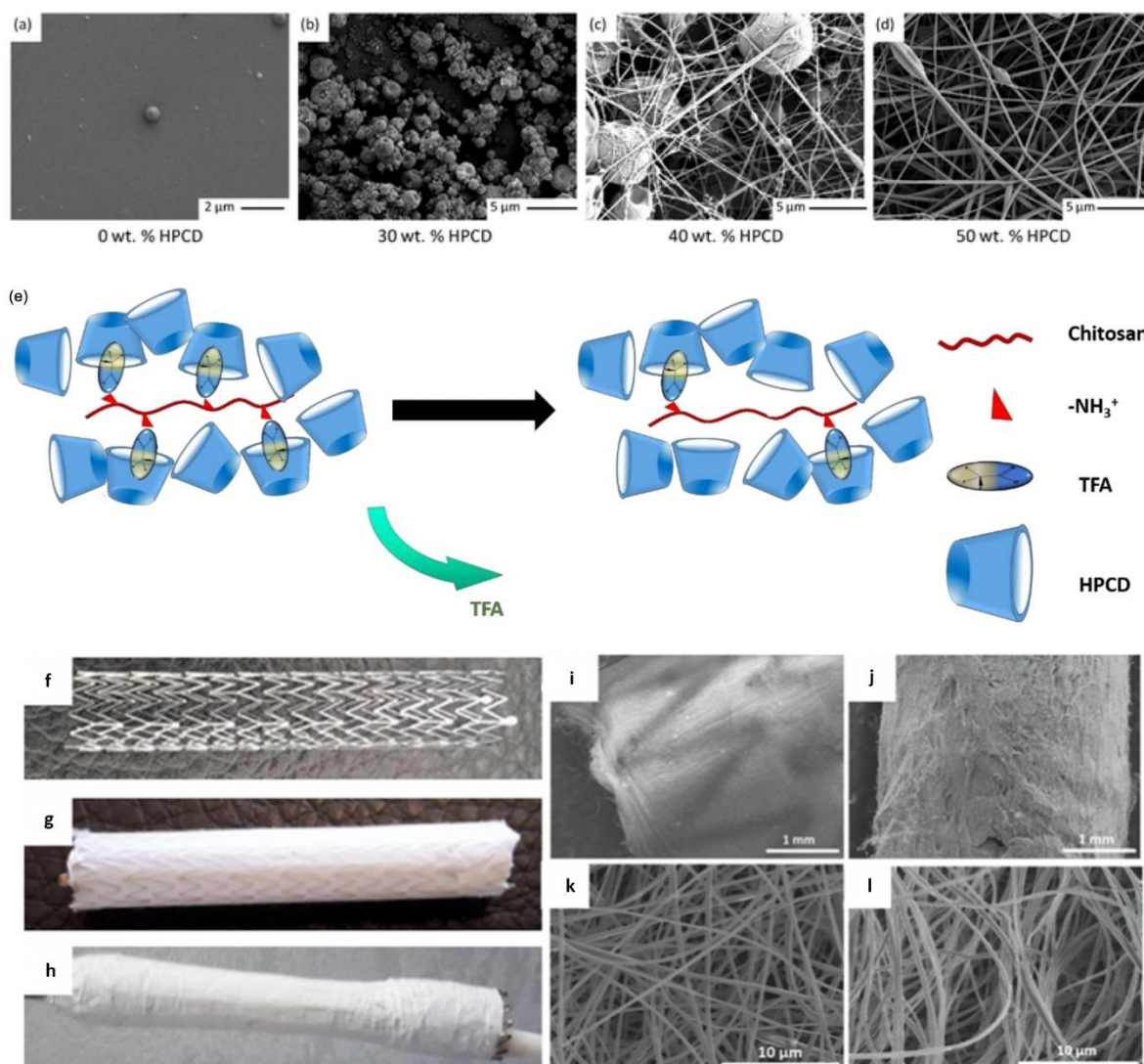
610 al. (Vicini et al., 2018) used a bath collector electrospinning to obtain crosslinked composite
611 alginate/hyaluronic acid/PEO membranes. Similarly, Ge et al. (Ge et al., 2012) and Huang et al. (X. J. Huang
612 et al., 2007) reported on chitosan/PVA electrospun membranes for the encapsulation of bioactive compounds,
613 whereas pullulan (PUL)/PVA nanofibers with UV-resistant properties were obtained by Qian et al. (Qian et
614 al., 2016). Compared to pure polysaccharide nanofibrous membranes, cyclodextrin-functionalized
615 polysaccharide nanofibers offer several advantages that will be deeply discussed in this section with a
616 complete and up-to-date summary of the literature reported in Table 2.

617 **Table 2.** Comprehensive summary of polysaccharide-based electrospun membranes functionalized with CDs or CD-ICs and their main applications.

Polysaccharide	Cyclodextrins	Encapsulated bioactive molecules	Application	Ref.
Chitosan	βCDs	---	Cyclodextrin grafted nanofibers for effluent dye removal	(Norouzi et al., 2012)
		Cinnamon and oregano essential oils	Nanofibrous membranes with strong antibacterial properties for biomedical and food packaging applications	(Munhuweyi et al., 2018)
	CMβCDs	Salicylic acid	Cyclodextrin grafted nanofibers as drug delivery systems with tuneable release kinetics	(Bazhban et al., 2013)
	HPβCDs	---	Fundamental study of the effect of CDs on chitosan solution spinnability	(Burns et al., 2015; C. Xue & Wilson, 2019)
	Poly-HPβCDs	Triclosan	Biomedical and food packaging products with improved water stability, tenable release kinetics and antibacterial properties	(Ouerghemmi et al., 2016)
		Simvastatin	Angioplasty bare metal stent covering layer for restenosis prevention	(Kersani et al., 2020)
Pullulan	βCDs	Limonene, Perillaldehyde	Edible food packaging products with antibacterial properties, high stability and humidity triggered release behaviour	(Fuenmayor et al., 2013; Mascheroni et al., 2013)
	γCDs	Eugenol	Biomedical and food packaging products with enhanced release and antioxidant properties	(Celebioglu & Uyar, 2020f)
Cellulose acetate	βCDs	N-halamine copolymers	Biomedical and food packaging products with biocidal properties	(R. Li et al., 2014)
		Ag/Fe-NPs	Filter materials embedding in-situ synthesized metal nanoparticles for wastewater purification	(Nthunya et al., 2017)
		---	Odour adsorbent filter materials for oil and fat derived volatile compounds	(Ghorani et al., 2019)
		---	Cyclodextrin grafted nanofibers for the removal of phenanthrene	(Celebioglu, Demirci, et al., 2014)
		---	Cyclodextrin grafted nanofibers as filter materials for toluene in wastewater	(Yuan et al., 2017)
		Tetrafluoroterephthalonitrile	Cyclodextrin grafted nanofibers for the removal of bisphenol pollutants	(Y. Lv et al., 2021)

		Thymol	Porous nanofibers with antibacterial properties and drug delivery capabilities for biomedical applications	(Yajun Chen et al., 2020)
	α CDs	---	Cyclodextrin grafted nanofibers as filter materials for toluene in wastewater	(Yuan et al., 2017)
	γ CDs	---	Cyclodextrin grafted nanofibers as filter materials for toluene in wastewater	(Yuan et al., 2017)
Hydroxypropyl cellulose	HP β CDs	Sulfisoxazole	Drug delivery systems with enhanced release abilities	(Aytac et al., 2015)
Almond gum	β CDs	Curcumin	Drug delivery systems with enhanced encapsulation capabilities for oral and gastrointestinal formulation dosages	(Rezaei & Nasirpour, 2019)
Hyaluronic acid	HP β CDs	Naproxen	Wound dressing patches with controlled drug delivery abilities enhanced water-stability	(Séon-Lutz et al., 2019)
Dandelion	β CDs	Litsea Cubeba essential oils	Membranes with strong antibacterial properties for biomedical and food packaging purposes	(Cui et al., 2019)

618 *Chitosan* (CH) is a polysaccharide derived from the partial deacetylation of chitin, the second most abundant
619 biopolymer in nature after cellulose and widely present in the exoskeleton of shellfish and fungi organisms.
620 Owing to its unique properties and wide availability, chitosan has been recognized as one of the most
621 promising materials of the future to substitute synthetic polymers (Riaz Rajoka et al., 2019). The first study
622 concerning chitosan nanofibers functionalized with cyclodextrins can be attributed to Norouzi et al. (Norouzi
623 et al., 2012) who reported on chitosan/PVA electrospun membranes grafted with β -cyclodextrins for the
624 efficient dye removal from effluents. After a crosslinking procedure carried out with glutaraldehyde vapours,
625 the prepared samples were able to retain their nanofibrous structure for a long time during which they
626 showed a surprisingly high uptake capacity. Similarly, Bazhban et al. (Bazhban et al., 2013) prepared
627 chitosan/PVA electrospun membranes grafted with carboxymethyl- β -cyclodextrins (CM β CDs) as drug
628 delivery systems demonstrating a slower release compared to the correspondent polymer films. Subsequently,
629 Burns et al. (Burns et al., 2015) tried to understand the effect of HP β CDs on the electrospinning process of
630 chitosan solutions in acetic acid (AA) and trifluoroacetic acid (TFA). The authors observed a strong
631 improvement in the processability of CH solutions enriched with HP β CDs with more homogeneous
632 nanofibers obtained by increasing the cyclodextrin concentration as shown in Figure 7a-d.



633

634 **Figure 7.** Fiber morphology of chitosan-based electrospun mats with an increasing HPβCD content: (a) 0
 635 wt%, (b) 30 wt%, (c) 40 wt%, and (d) 50 wt%. Reproduced with permission from Burns et al., 2015. © The
 636 Royal Society of Chemistry 2015. (e) Schematic illustration of CH/HPβCD electrospun nanofiber
 637 composition. Reproduced with permission from C. Xue & Wilson, 2019. (f) Macroscopic and microscopic
 638 images of Nitinol stent covered with CH/polyHPβCD-ICs-simvastatin nanofibers before (g, i, k) and after
 639 crimping (h, j, l). Reproduced with permission from Kersani et al., 2020. © 2020 Elsevier B.V.

640

641 Such findings were attributed to the formation of a stable inclusion complex between the hydrophobic cavity
 642 of cyclodextrins and chitosan amine groups. More recently, Xue et al. (C. Xue & Wilson, 2019) carried out a
 643 solid-state spectroscopic and thermal study on CH/HPβCD electrospun nanofibers obtained using TFA as
 644 solvent. On the contrary to what reported by Burns et al. (Burns et al., 2015), it was suggested that the
 645 enhanced processability of chitosan solutions was likewise related to the formation of a ternary complex
 646 between CH, HPβCDs, and TFA with the solvent molecules acting as bridges, as schematically depicted in
 647 Figure 7e. By Raman imaging, the authors were able to prove the absence of direct interactions between

648 chitosan and cyclodextrins opening the way to the development of new supramolecular structures. Along
649 with these preliminary studies, Ouerghemmi et al. (Ouerghemmi et al., 2016) prepared chitosan electrospun
650 membranes containing the inclusion complexes between polyHP β CDs and triclosan. Compared to pristine
651 HP β CDs, polyHP β CDs provided a higher encapsulation capability and led to the formation of a
652 polyelectrolyte complex with chitosan enhancing the water-resistance to the nanofibers. With respect to
653 chitosan nanofibers functionalized with HP β CD-ICs-TCL or free TCL, CH/polyHP β CD-ICs-TCL mats
654 showed a slower release over a longer period both at pH = 7.4 and pH = 5.5 conditions. Moreover, the
655 authors proved that the mats were embedded with strong antibacterial activities remarkably higher in terms
656 of growth inhibition zone compared to those of similar synthetic products (Kayaci, Umu, et al., 2013; Tonelli,
657 2008), thus making them suitable candidates for both biomedical and food packaging applications. Yet,
658 Munhuweyi et al. (Munhuweyi et al., 2018) reported on the encapsulation of cinnamon and oregano essential
659 oils in CH/PVA nanofibers embedding β CDs. Owing to the great encapsulation efficiency, which was much
660 higher compared to PLA-, PVA-, and PCL-based nanofibers containing cinnamon oil (Amiri & Rahimi,
661 2019; Pan et al., 2019; Wen, Zhu, Feng, et al., 2016; Wen, Zhu, Wu, et al., 2016), the proposed
662 CH/PVA/ β CD-ICs-cinnamon mats showed particularly marked antibacterial properties against *Botrytis* sp.
663 bacteria, hence representing interesting materials especially for food packaging applications. Recently,
664 Kersani et al. (Kersani et al., 2020) developed a bare-metal stent for angioplasty covered by an electrospun
665 layer of chitosan/polyHP β CD-ICs-simvastatin (SV) drug as an efficient tool for restenosis prevention
666 represented in Figure 7a. Compared to the nanofibers loaded with free SV or embedding HP β CD-ICs-SV,
667 the use of polyHP β CDs highly improved the drug loading efficiency leading to a much more prolonged and
668 controlled release from the mats. Additionally, the release of simvastatin was found to be tuneable by simply
669 changing the sample thickness or the initial loading concentration indicating the possibility to accurately
670 personalize the system depending on the patient requirements.

671 *Pullulan* (PUL) is a water-soluble extracellular polysaccharide obtained from fermentation processes and
672 exploited in the manufacturing of edible films, as well as in several biomedical and pharmaceutical oral care
673 applications (Prajapati et al., 2013). In this regard, Fuenmayor et al. (Fuenmayor et al., 2013) developed
674 edible nanofibrous membranes by single-step electrospinning of a pullulan/ β CD aqueous solution containing

675 R-(+)-limonene, which was found to be stable over a long time with its release occurring only at high water
676 activity values (a_w) hence making the prepared mats suitable for fresh food packaging products. Similarly,
677 Mascheroni et al. (Mascheroni et al., 2013) investigated the encapsulation of the volatile compound
678 perillaldehyde, an aroma largely employed in the food industry, within a pullulan electrospun matrix
679 containing β CDs. No evident losses of the aroma compound were observed over several months at ambient
680 conditions with the release occurring exclusively for $a_w > 0.9$. Lately, Celebioglu et al. (Celebioglu & Uyar,
681 2020f) reported on pullulan nanofibers embedding γ CD-ICs-eugenol able to almost preserve the volatile
682 compound with an encapsulation efficiency of $\sim 93\%$, which was considerably higher with respect to
683 pullulan/eugenol nanofibers (i.e., $\sim 23\%$). Moreover, the complexation increased the thermal stability of
684 eugenol, whereas its antioxidant properties were substantially enhanced and remained unvaried over a period
685 of 3 months.

686 *Cellulose acetate* (CA) is one of the most important derivatives of cellulose and it is widely employed as a
687 film base for photography, vapour sensors, optical materials, and to produce synthetic fibers for textile
688 applications owing to its renewable source, biodegradability, non-toxicity and low cost (Candido &
689 Gonçalves, 2016; Lova et al., 2018, 2019; Ummartyotin & Manuspiya, 2015). Li et al. (R. Li et al., 2014)
690 prepared CA nanofibrous membranes embedding β CD inclusion complexes with an ad hoc synthesized N-
691 halamine copolymer as promising candidates for biomedical and food packaging applications with strong
692 biocidal properties against both Gram-positive and Gram-negative bacteria. Aiming to the efficient removal
693 of bacteria from drinking water, Nthunya et al. (Nthunya et al., 2017) investigated the possibility to prepare
694 cellulose acetate/ β -cyclodextrin membranes embedding in-situ synthesized metal nanoparticles. Ag/Fe
695 nanoparticles were here prepared via a green UV photoreduction process under ambient conditions and
696 enriched the mats with strong antibacterial properties against several bacteria strains thus making them a
697 valid alternative for water purification purposes. More recently, Ghorani et al. (Ghorani et al., 2019)
698 developed CA nanofibers embedding β CDs as odour adsorbent filter materials by selecting acetone:DMF
699 (2:1) as the most appropriate solvent system to obtain a considerably high production rate suitable for
700 industrial applications. Above all, the proficiency of the prepared mats to adsorb hexanal molecules, which
701 are a marker for oil and fat oxidation, suggested their suitability for filtration purposes. While the previously
702 mentioned works consist in the direct electrospinning of CA/CD solutions, a different approach was first

703 proposed by Celebioglu et al. (Celebioglu, Demirci, et al., 2014) who reported on cellulose acetate
704 nanofibers grafted with β CDs via a simple “click” reaction carried out to avoid leaking issues. The grafted
705 membranes were tested as potential molecular filters for water purification purposes showing greater
706 adsorption capability in removing phenanthrene compared to the pristine CA nanofibers, as well as respect to
707 comparable PET/CD nanofibers (Kayaci, Aytac, et al., 2013). Similarly, Yuan et al. (Yuan et al., 2017)
708 investigated the possibility to use regenerated cellulose (RC) nanofibers functionalized with α CDs, β CDs
709 or γ CDs via physical mixing or grafting reaction for the removal of toluene residues from wastewater. Each
710 type of CD led to homogenous nanofibers with the grafting approach enriching the mats with considerably
711 higher adsorption capability compared to the works of Kayaci et al. (Kayaci et al., 2015) and Uyar et al.
712 (Uyar et al., 2010). Yet, Chen et al. (Yajun Chen et al., 2020) reported on the development of hierarchical
713 CA nanofibers embedding the inclusion complex between β CDs and thymol. The proposed membranes
714 showed a prolonged release resulting in marked antibacterial properties, mechanical properties suitable for
715 wound dressing applications, and excellent cytocompatibility. Lately, Lv et al. (Y. Lv et al., 2021) prepared
716 β -cyclodextrin cellulose acetate nanofibers for the removal of bisphenol pollutants from water. The authors
717 first modified native β CDs with tetrafluoroterephthalonitrile (TF) by a simple chemical treatment before
718 grafting them onto the CA mats to enhance the pollutant adsorption efficiency. The obtained products
719 showed a highly porous morphology, a stable crystal structure, and excellent thermal stability, as well as the
720 abundance of surface functional groups. Noticeably, the authors demonstrated the capability of the proposed
721 nanofibers to efficiently act as adsorbent materials in a broad range of conditions for several cycles
722 indicating their suitability for practical applications.

723 *Hydroxypropyl cellulose* (HPC) is another cellulose derivative showing good solubility in both water and
724 organic solvents and mainly used as an excipient, lubricant and ophthalmic protectant owing to its thermo-
725 and pH-responsive properties (Qiu & Hu, 2013; Yang & Li, 2018). In this regard, Aytac et al. (Aytac et al.,
726 2015) presented the preparation of HPC nanofibers functionalized with the inclusion complex between
727 hydroxypropyl- β -cyclodextrin and sulfisoxazole (SFS) for drug delivery applications by developing
728 sandwich-like multilayer materials. Compared to nanofibers containing the free drug, HPC/HP β CD-ICs-SFS

729 mats showed a considerably higher release most likely owing to the enhanced incorporation of SFS through
730 inclusion complexation.

731 *Almond gum* (ALG) is a natural gum copiously exuded from the trunk, branches, and fruits of *Prunus*
732 *amygdalus* trees after mechanical injury and/or infection by microorganisms. Almond gum represents an
733 important waste product in several developing countries and, owing to its antibacterial and antioxidant
734 activities, could be successfully employed as a green alternative to several synthetic materials (Bouaziz et al.,
735 2017). The release mechanism of curcumin from almond gum/PVA/ β CD-ICs-curcumin electrospun
736 membranes in simulated saliva and gastrointestinal conditions was investigated by Rezaei et al. (Rezaei &
737 Nasirpour, 2019). Compared to the nanofibers loaded with free curcumin, the presence of β CD-curcumin ICs
738 allowed to reach a much higher compound encapsulation efficiency (i.e., > 90%) and enriched the
739 membranes with a greater and faster release, thus proving the possibility to use almond gum-based
740 composite nanofibers as efficient drug delivery systems.

741 *Hyaluronic acid* (HA) is an anionic polysaccharide present in several human tissues with unique physical-
742 chemical properties and a wide range of important biological functions, including cell signalling, wound
743 repair, tissue regeneration, morphogenesis, matrix organization and pathobiology (Dodero, Williams, et
744 al., 2019; Fallacara et al., 2018). Séon-Lutz et al. (Séon-Lutz et al., 2019) reported on the preparation of HA-
745 PVA membranes functionalized with HP β CDs for wound dressing applications using only water as solvent.
746 The membranes were thereafter crosslinked with a carbodiimide and N-hydroxysuccinimide ensuring thus a
747 completely green process of fabrication. The authors proved that the use of HP β CDs stabilized the
748 electrospinning process owing to their capability to establish intermolecular interactions with HA and PVA,
749 thus allowing to noticeably reduce the minimum concentration of co-spinning agent (i.e., PVA). More
750 importantly, naproxen (NAP) drug was a posteriori encapsulated within the prepared nanofibers and
751 presented a sustained release over several days.

752 *Dandelion* (DP) is an α -type polysaccharide extracted from the roots of a perennial herbaceous plant and
753 currently employed in several food products owing to its antitumor, antioxidant, and anti-complement
754 properties (Guo et al., 2019). Cui et al. (Cui et al., 2019) reported on the preparation of nanofibrous
755 dandelion-based membranes embedding β CD-ICs-Litsea cubeba essential oil (LCEO) as efficient
756 antibacterial agents. The structure of the mats was improved thanks to the addition of the β CD complexes in

757 the spinning solutions owing to the interactions between the components. Interestingly, a sustained release of
758 the encapsulated LCEO was observed over several days resulting in marked antibacterial properties,
759 therefore making the prepared nanofibers suitable to be used for both biomedical and food packaging
760 purposes.

761

762 **6.2 Electrospun Protein – Cyclodextrin Based Membranes**

763 Proteins are biomacromolecules composed of long amino acid chains with an important role in several body
764 functions such as DNA replication, metabolic reactions, stimuli responses, and moieties transportation, as
765 well as in cell viability. Proteins are biocompatible, biodegradable, easily accustomed in the human body,
766 and with interesting isoelectric properties that allow them to interact with a wide variety of molecules
767 (Muiznieks & Keeley, 2017; Sheng Wang et al., 2016; Weisel & Litvinov, 2017). Owing to such properties,
768 it is not surprising that protein-based electrospun membranes are gaining increasing interest for several
769 applications, especially in the food packaging and biomedical industries. However, proteins are known to be
770 infamously difficult to be electrospun because of their complex secondary and ternary structures, with
771 globular properties having too few interactions to form a sufficient number of entanglements (Babitha et al.,
772 2017; DeFrates et al., 2018; Ding et al., 2019; Kakoria & Sinha-Ray, 2018; Zhong et al., 2018).

773 Protein-based nanofibrous membranes functionalized with cyclodextrins represent an emerging and
774 interesting class of novel materials with the possibility to couple the outstanding properties of both
775 components. However, their investigation remains to an initial stage with only a small number of
776 publications reported in the literature as summarized in Table 3.

Table 3. Comprehensive summary of protein-based electrospun membranes functionalized with CDs or CD-ICs and their main applications.

Protein	Cyclodextrins	Encapsulated bioactive molecules	Applications	Ref.
Soy protein isolate	β CDs	Allyl isothiocyanate	Food packaging materials with enhanced encapsulation efficiency and humidity triggered release kinetic	(Vega-Lugo & Lim, 2009)
	α CDs	---	Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)
Zein	β CDs	---	Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)
		Eucalyptus essential oil	Antibacterial food packaging products with enhanced thermal stability	(Dias Antunes et al., 2017)
	Quercetin	Antibacterial food packaging products	(Z. Wang et al., 2021)	
	---	Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)	
	γ CDs	Thymol	Antibacterial food packaging products with enhanced release properties	(Aytac, Ipek, et al., 2017)
		Quercetin	Water-insoluble food packaging materials with fast antioxidant action	(Aytac, Ipek, et al., 2018)
Silk fibroin	HP β CDs	Tamoxifen	Transdermal drug delivery systems with prolonged-release kinetic for the treatment of breast cancer	(W. Liu et al., 2016)
Gelatin	β CDs	---	Water-insoluble crosslinked nanofibers as cost-effective water pollutant removal	(Yu Chen et al., 2018)
		---	Respiratory filter media for filtration of aerosols and volatile organic compounds	(Kadam et al., 2021)
		Galangal root oil	Active food packaging products with antibacterial properties	(Zhou et al., 2020)
	HP β CDs	Ciprofloxacin	Fast-dissolving drug delivery systems for oral formulation dosages	(Aytac et al., 2019)
Gliadin	HP β CDs	Ferulic acid	Biomedical and food products with enhanced thermal stability and fast release properties	(Sharif et al., 2018)
Hordein	β CDs	N,N'-methylene bisacrylamide	Recyclable filter materials for the efficient removal of heavy metals from wastewater	(Guan et al., 2019)

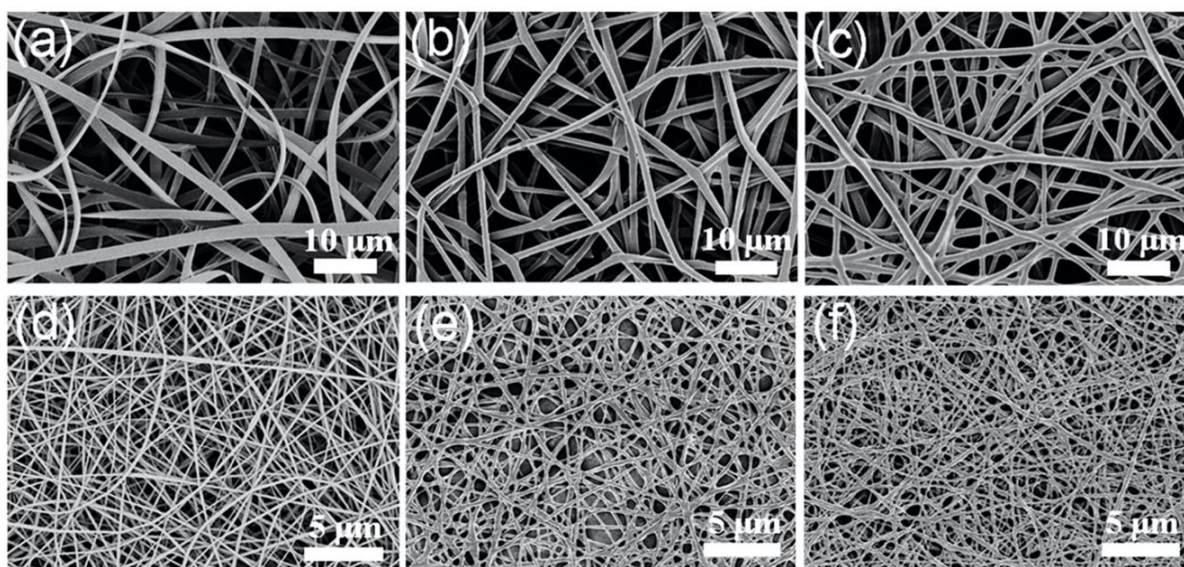
778 *Soy protein isolate* (SPI) is the most refined protein obtained from soybean with several applications in
779 food-related products due to its great abundance and high availability as by-products from industries (Rani &
780 Kumar, 2019; Tian et al., 2018). SPI was the first protein employed to prepare protein-based electrospun
781 nanofibers functionalized with cyclodextrins by Vega-Lugo et al. (Vega-Lugo & Lim, 2009), who reported
782 on SPI nanofibers encapsulating allyl isothiocyanate compound (AITC) by using β CDs as complexation
783 agents and PEO as a co-spinning agent. Beyond the possibility to tune the nanofiber structure by simple
784 changing the mixture composition, AITC release kinetics from the composite nanofibers strongly depended
785 on the environment relative humidity. Specifically, no compound loss was observed at 0% RH, whereas a
786 sustained release for 20h was detected at 75% RH comparably to the results showed by Aytac et al.
787 concerning PVA/ β CD-ICs-AITC membranes (Aytac et al., 2014).

788 *Zein* (ZN) is a clear, odourless, tasteless, water-insoluble and edible prolamin protein derived from corn and
789 broadly employed in several commercial products ranging from the textile fiber market to the food industry
790 (Kasaai, 2017; Turasan & Kokini, 2017; Yong Zhang et al., 2016). Kayaci et al. (Kayaci & Uyar, 2012a) first
791 reported on a fundamental study regarding the preparation of zein nanofibers encapsulating different amount
792 of α CDs, β CDs, or γ CDs. Comparably to what was reported by other research groups for polysaccharides,
793 the addition of CDs noticeably improved the spinnability of pristine zein solutions leading to bead-free
794 nanofibers, with only a slight morphological dependence on the cyclodextrin type, and enhanced thermal
795 stability. Owing to the abovementioned finding and keeping in mind cyclodextrin complexation capability,
796 the authors indicated that these nanofibrous mats are promising products for biomedical and food packaging
797 applications. Subsequently, Aytac et al. (Aytac, Ipek, et al., 2017) prepared zein nanofibers functionalized
798 with the inclusion complexes between γ -cyclodextrins and thymol, which were able to provide a high
799 encapsulation capability leading to enhanced stability and release kinetic. Moreover, by testing the
800 antibacterial properties toward *E. Coli* and *S. Aureus* bacteria in meat simulated storage conditions at T =
801 4 °C, the prepared mats were suggested as promising antibacterial food packaging materials. Similar work
802 was reported by Antunes et al. (Dias Antunes et al., 2017), who proposed the encapsulation of β -
803 cyclodextrin-eucalyptus essential oil complexes in zein electrospun nanofibers by using aqueous ethanol
804 (EtOH) eco-friendly mixture as solvent. SEM images proved the good homogeneity of the obtained
805 nanofibers, which contained around 24% of the formed inclusion complexes. Moreover, the antibacterial

806 properties were tested against *S. aureus* and *Listeria monocytogenes* (*L. monocytogenes*) bacteria showing a
807 strong growth reduction and the possibility to use the obtained mats for applications in the food packaging.
808 More recently, Aytac et al. (Aytac, Ipek, et al., 2018) reported on electrospun zein/ γ CD-ICs-quercetin
809 nanofibers as quick and efficient antioxidant materials, with the encapsulated quercetin showing enhanced
810 thermal stability and water solubility compared to its free form. Similarly, Wang et al. (Z. Wang et al., 2021)
811 prepared homogenous and free-standing zein nanofibers embedding β CD-ICs-quercetin. The encapsulation
812 of the bioactive compound was proved to improve its availability, release time, and thermal stability.
813 Consequently, the obtained mats showed strong antibacterial properties against both *E. Coli* and *S. Aureus*
814 bacteria, thus resulting in promising materials for future active food packaging products.

815 *Silk fibroin* (SF) is an insoluble protein largely produced by the larvae of *Bombyx mori* and numerous other
816 insects. SF has assumed an increasing interest in biomedical applications owing to its biocompatibility, low
817 immunogenicity, mechanical properties, and ability to remodel in response to the biological environment
818 (Koh et al., 2018; Qi et al., 2017). With this premises, Liu et al. (W. Liu et al., 2016) demonstrated the
819 possibility to prepare regenerated silk fibroin (RSF) nanofibers embedding HP β CD-ICs-tamoxifen (TAM) as
820 potential drug delivery systems by using a simple aqueous mixture. The complex addition strongly increased
821 the spinnability of RSF solutions leading to thinner and more homogenous nanofibers as shown in Figure 8,
822 whereas the encapsulated drug was slowly released over 3 weeks making RSF cyclodextrin-functionalized
823 nanofibers promising products as transdermal drug delivery systems.

824



825

826 **Figure 8.** SEM micrographs of (a) as-spun RSF nanofibers, (b) RSF nanofibers after ethanol spraying
 827 treatment, (c) RSF nanofibers after acetone immersion treatment, (d) as-spun RSF/HPβCD-ICs-TAM
 828 nanofibers, (e) RSF/HPβCD-ICs-TAM nanofibers after ethanol spraying treatment, (f) RSF/HPβCDs-ICs-
 829 TAM nanofibers after acetone immersion treatment. Reproduced with permission from W. Liu et al., 2016.
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831

832 *Gelatin* (GL) is a largely available protein obtained from animal products and broadly employed in
 833 manufacturing to prepare foods, cosmetics and medicines owing to its gelling properties and ability to
 834 improve product elasticity, consistency and stability (Hosseini & Gómez-Guillén, 2018; Karayannakidis &
 835 Zotos, 2016). The use of gelatin/β-cyclodextrins nanofibers for the removal of dyes from wastewater was
 836 proposed by Chen et al. (Yu Chen et al., 2018). The authors developed a crosslinking approach with
 837 glutaraldehyde vapour both to stabilize the nanofibers and permanently grafting the cyclodextrins to the
 838 polymer chains avoiding leaking issues. Remarkably, the prepared mats showed promising adsorption
 839 capability towards methylene blue in different experimental conditions and several other common dyes,
 840 being above all characterized by good recyclability and reusability, therefore representing cost-effective and
 841 promising products for the efficient purification of wastewater. Yet, Aytac et al. (Aytac et al., 2019)
 842 investigated the use of gelatin nanofibers loaded with HPβCD and ciprofloxacin (CIP) complexes as fast-
 843 dissolving drug delivery systems. The authors were able to prove that the bead-free nanofibers encapsulating
 844 HPβCD-ICs-CIP showed to be more rapidly dissolved compared to gelatin/CIP nanofibers, thus pointing out
 845 the capability of HPβCDs in increasing the sample wettability and opening the way to the preparation of a
 846 broad variety of drug delivery systems able to quickly release high drug dosages. Additionally, Zhou et al.

847 (Zhou et al., 2020) investigated the preparation of gelatin-based nanofibers containing the inclusion complex
848 between β CDs and galangal root oil (GRO). By using different gelatin sources, the authors evaluated how
849 the composition of the protein amino acids affected the electrospinning process. Remarkably, gelatin/ β CD-
850 ICs-GRO showed marked antibacterial properties against E Coli, as well as excellent mechanical and thermal
851 properties, thus being suitable as food packaging materials. Lately, Kadam et al. (Kadam et al., 2021)
852 reported on the preparation of gelatin/ β CDs nanofibers to be used as filter materials. In particular, the
853 possibility to easily control the fiber dimension and the mat porosity, coupled with the cyclodextrin inclusion
854 complex capabilities, allowed the prepared materials to show filtration efficiency towards both solid/liquid
855 particles and VOCs.

856 *Gliadin* (GD) is a prolamin protein largely present in wheat, as well as in other cereals, with a wide range of
857 interesting applications (Jia et al., 2020; Urade et al., 2018). Sharif et al. (Sharif et al., 2018) reported on the
858 encapsulation of HP β CD-ICs-ferulic acid within gliadin electrospun nanofibers. The complexation
859 phenomenon allowed to increase both the thermal and photostability of FA thus preserving its antioxidant
860 properties. Noticeably, compared to the GL nanofibers containing free FA, gliadin/HP β CD-ICs-FA mats
861 showed a superior and faster release of the bioactive compound in artificial media simulating different food
862 or biological conditions.

863 *Hordein* (HD), likewise gliadin, is a prolamin protein present in barley and other cereals and mainly used for
864 food applications (X. Huang et al., 2017). Guan et al. (Guan et al., 2019) proposed hordein nanofibrous
865 membranes embedding N,N'-methylene bisacrylamide (MBA) molecules and grafted with β CDs for the
866 adsorption of copper ions. The uptake properties of the prepared mats were evaluated at different pH and
867 temperature conditions with the hordein/ β CD-ICs-MBA nanofibers showing higher adsorption capability
868 both compared to hordein/MBA and hordein electrospun mats. Additionally, the authors were able to prove
869 the recyclability of the obtained mats via a humble and cost-effective washing procedure hence
870 demonstrating their efficient use for the treatment of wastewater.

871

872 **7 Conclusions and Future Perspectives**

873 Nowadays, electrospinning is worldwide recognized as a simple, flexible, and cost-effective technique for
874 the preparation of nano- and microfibers with applicability in almost any research field and it has become, in
875 the last decade, one of the most promising approaches to improve human quality of life via the production of
876 tailored-made products for biomedical, food, energy harvesting, textile and environmental purposes. In this
877 sense, cyclodextrins may play a very important role and their use in combination with electrospinning has
878 aroused growing interest. Specifically, in most cases, the objective is to prepare polymer-free cyclodextrin
879 nanofibers able to efficiently encapsulate bioactive compounds and accurately control their release. Indeed,
880 the outstanding capability of cyclodextrins to form non-covalent stable complexes with small molecules (e.g.,
881 hydrophobic and/or volatile compounds) allows to considerably increase their solubility, stability, and
882 availability, hence leading to a superior encapsulation efficiency that was previously unreachable. However,
883 a variety of innovative applications are recently being evaluated for electrospun nanofibers made of (or
884 containing) cyclodextrins and their derivatives, also creditable to the fact that their preparation allows
885 avoiding the intensive use of toxic solvents and/or hazardous chemicals. For instance, cyclodextrin-based
886 nanofibers can be employed as reducing agents for the in-situ synthesis of metal nanoparticles, as sacrificial
887 templates for the development of conductive hollow structures, as implantable scaffolds for tissue
888 regeneration purposes, as well as to develop high performing filtering materials and food packaging products
889 with antibacterial and antioxidant properties.

890 Additionally, combining the complexation capability of cyclodextrins with naturally occurring biopolymers
891 appears to be the optimal format for the development of mats with unique and tuneable features. For instance,
892 besides biocompatibility and greater biological response, biodegradability and digestibility, the greatest
893 advantage of natural biopolymers is that they can be processed by using water or poorly toxic organic
894 solvents making them extremely interesting from an industrial point of view. Moreover, the continuous
895 advances in the extraction of materials from natural waste sources may lead in the future to the development
896 of environmentally friendly electrospun products in view of a green circular economy. However, the
897 development of these strategies remains at an initial stage and further studies are required since, at present,
898 the electrospinning of natural biopolymers requires appropriate additives to facilitate the processing, as well
899 as post-processing treatments to increase the mat stability and mechanical performances without affecting the

900 intrinsic biocompatibility. Noticeably, it is noteworthy that cyclodextrins have been demonstrated able to
901 enhance the spinnability of biopolymer-based formulations due to the tendency to create supramolecular
902 structures with the biopolymers, thus reducing, or even eliminating, the amount of required synthetic co-
903 spinning agents. Nevertheless, despite the considerable advances recently made at the laboratory scale, the
904 industrial implementation of natural polymer electrospinning has been scarcely explored and remains a great
905 challenge especially in terms of production rate.

906 In conclusion, in order to ensure the efficient translation between laboratory prototypes and commercial
907 products, workability studies are mandatory to verify the effective applicability of cyclodextrin-based
908 electrospun membranes for the proposed applications and more research-industry collaborations are
909 indispensable to lower the cost and improve the industrialization of such materials.

910

911 **Conflict of Competing Interest**

912 None.

913

914 **Funding**

915 This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-
916 profit sectors.

917 **Abbreviations**

918	AA	Acetic acid
919	AAB	Azobenzene
920	AFM	Atomic force microscopy
921	Ag	Silver
922	Ag-NPs	Silver nanoparticles
923	AITC	Allyl isothiocyanate compound
924	Al	Aluminium
925	Al ₂ O ₃	Aluminium oxide
926	ALD	Atomic layer deposition
927	ALG	Almond gum
928	Au	Gold
929	Au-NPs	Gold nanoparticles
930	a _w	Water activity
931	BA	Benzoxazine
932	BET	Brunauer – Emmett – Teller
933	Botrytis sp.	Botrytis species
934	BTCA	Butanetetracarboxylic acid
935	CA	Cellulose acetate
936	CTR	Citric acid
937	CD-ICs	Cyclodextrin inclusion complexes
938	CDs	Cyclodextrins
939	c _e	Entanglement concentration
940	CH	Chitosan
941	CIP	Ciprofloxacin
942	CMβCDs	Carboxymethyl-β-cyclodextrins
943	COL	Collagen
944	Cr	Chromium

945	Cu	Copper
946	DCF	Diclofenac sodium
947	DDSs	Drug delivery systems
948	DLS	Dynamic light scattering
949	DMA	Dynamic-mechanical analysis
950	DMAc	Dimethylacetamide
951	DMF	Dimethylformamide
952	DMSO	Dimethyl sulfoxide
953	DNA	Deoxyribonucleic acid
954	DOX	Doxycycline
955	DP	Dandelion
956	DSC	Differential scanning calorimetry
957	E. Coli	Escherichia Coli
958	ECH	Epichlorohydrin
959	ECM	Extra-cellular matrix
960	EDS	Energy-dispersive X-ray spectroscopy
961	ES	Electrospinning
962	EtOH	Ethanol
963	FA	Ferulic acid
964	Fe	Iron
965	Fe-NPs	Iron nanoparticles
966	FTIR	Fourier-transform infrared spectroscopy
967	GD	Gliadin
968	GL	Gelatin
969	GRO	Galangal root oil
970	HA	Hyaluronic acid
971	HD	Hordein
972	HPC	Hydroxypropyl cellulose

973	HP α CDs	2-hydroxypropyl- α CDs
974	HP β CDs	2-hydroxypropyl- β CDs
975	HP γ CDs	2-hydroxypropyl- γ CDs
976	HSES	High speed electrospinning
977	IKAV	Adamantane-6-aminohexanoic acid-GGKIKVAV-Am
978	KK	Adamantane-6-aminohexanoic acid-GGKK-Am
979	<i>L. monocytogenes</i>	<i>Listeria monocytogenes</i>
980	LCEO	Litsea cubeba essential oil
981	MB	Methylene blue
982	MBA	N,N'-methylene bisacrylamide
983	MO	Methyl orange
984	M β CDs	Randomly methylated- β CDs
985	NaOH	Sodium hydroxide
986	NAP	Naproxen
987	Ni	Nickel
988	NMR	Nuclear magnetic resonance spectroscopy
989	NPs	Nanoparticles
990	PAA	Poly(acrylic acid)
991	PAN	Polyacrylonitrile
992	PAP	p-aminophenol
993	PCL	Polycaprolactone
994	Pd	Palladium
995	Pd-NPs	Palladium nanoparticles
996	PEO	Poly(ethylene oxide)
997	PET	Polyethylene terephthalate
998	PHAs	Polycyclic aromatic hydrocarbons
999	PLA	Poly(lactic acid)
1000	PMMA	Polymethylmethacrylate

1001	PNP	p-nitrophenol
1002	PolyBA-a	Poly benzoxazine
1003	PolyCDs	Cyclodextrin-based polymers
1004	PolyHP β CDs	Poly-2-hydroxypropyl- β CDs
1005	PS	Polystyrene
1006	PUL	Pullulan
1007	PVA	Polyvinyl alcohol
1008	PVP	Polyvinylpyrrolidone
1009	RC	Regenerated cellulose
1010	RH	Relative humidity
1011	RSF	Regenerated silk fibroin
1012	S. Aureus	Staphylococcus Aureus
1013	SA	Sodium alginate
1014	SEB β CDs	Sulfobutylether- β -cyclodextrins
1015	SEM	Scanning electron microscopy
1016	SF	Silk fibroin
1017	SFS	Sulfisoxazole
1018	SHP	Sodium hypophosphite
1019	SPI	Soy protein isolate
1020	SV	Simvastatin
1021	SBG	Swim bladder gelatin
1022	TAM	Tamoxifen
1023	TBZ	Thiabendazole
1024	TCL	Triclosan
1025	TEM	Transmission electron microscopy
1026	TF	Tetrafluoroterephthalonitrile
1027	TFA	Trifluoroacetic acid
1028	TGA	Thermogravimetric analysis

1029	THY	Thymol
1030	UV	Ultraviolet
1031	UV-vis	Ultraviolet-visible
1032	VB ₂	Vitamin B ₂
1033	VOCs	Volatile organic compounds
1034	VOR	Voriconazole
1035	WAXS	Wide-angle X-ray scattering
1036	XPS	X-ray photoelectron spectroscopy
1037	XRD	X-ray diffraction
1038	ZN	Zein
1039	Zn	Zinc
1040	ZnO	Zinc oxide
1041	αCDs	α-cyclodextrins
1042	α-LA	alpha-lipoic acid
1043	βCDs	β-cyclodextrins
1044	γCDs	γ-cyclodextrin

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