

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

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1	Polymer-free Cyclodextrin and Natural Polymer-Cyclodextrin Electrospun Nanofibers: A
2	Comprehensive Review on Current Applications and Future Perspectives
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14	Abstract
15	The present review discusses the use of cyclodextrins and their derivatives to prepare electrospun nanofibers
16	with specific features. Cyclodextrins, owing to their unique capability to form inclusion complexes with
17	hydrophobic and volatile molecules, can indeed facilitate the encapsulation of bioactive compounds in
18	electrospun nanofibers allowing fast-dissolving products for food, biomedical, and pharmaceutical purposes,
19	filtering materials for wastewater and air purification, as well as a variety of other technological applications.
20	Additionally, cyclodextrins can improve the processability of naturally occurring biopolymers helping the
21	fabrication of "green" materials with a strong industrial relevance. Hence, this review provides a
22	comprehensive state-of-the-art of different cyclodextrins-based nanofibers including those made of pure
23	cyclodextrins, of polycyclodextrins, and those made of natural biopolymer functionalized with cyclodextrins.
24	To this end, the advantages and disadvantages of such approaches and their possible applications are
25	investigated along with the current limitations in the exploitation of electrospinning at the industrial level.
26	
27	Keywords

- 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 & Uvar, 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).

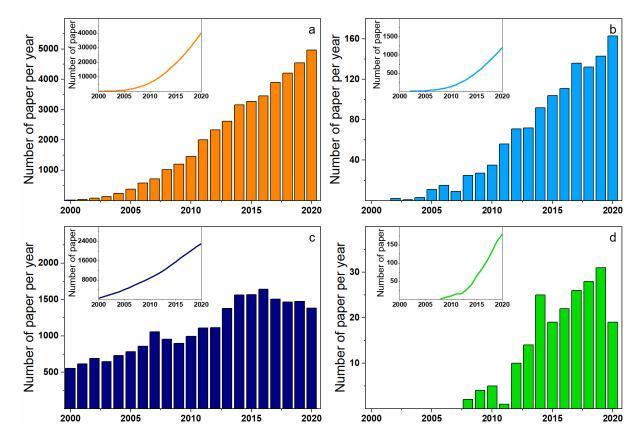


Figure 1. Annual trend of publications regarding (a) electrospinning, (b) functionalization of electrospun nanofibers, (c) cyclodextrins, and (d) cyclodextrin-functionalized electrospun nanofibers. Insets represent the cumulative number of publications. Data has been retrieved from Scopus database.

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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.

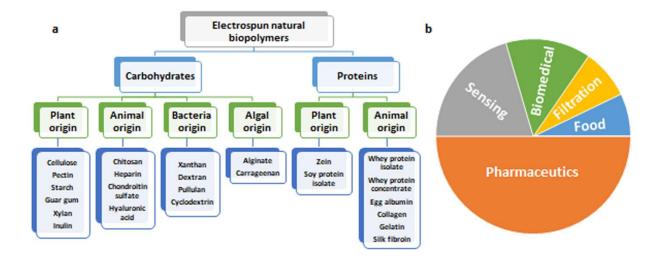


Figure 2. (a) Schematic of natural biopolymer-based electrospun membranes depending on the source
 materials and (b) pie chart representing their main applications according to the Scopus database.

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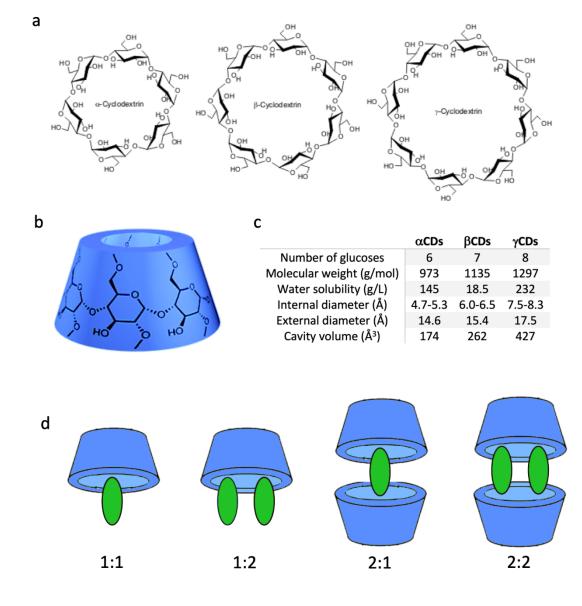
With these premises, aiming to highlight the importance of developing environmentally benign materials, this review aims to provide a comprehensive summary of the recent advances in the preparation of either polymer-free cyclodextrin-based nanofibers, polycyclodextrin-based nanofibers, or natural polymer-based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

75

76 2 Cyclodextrins

77 2.1 Structure and Properties

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



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

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 βCDs 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-βCDs (MβCDs), 2-hydroxypropyl-βCDs 99 (HPβCDs) and 2-hydroxypropyl-γCDs (HPγCDs) (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

The electrospinning (ES) technique consists of the application of a high voltage to a polymer solution, or a polymer melt, that is usually extruded from a spinneret at a constant rate. Owing to the presence of free surface charges, the polymeric droplet exiting the spinneret takes the shape of a cone (i.e., Taylor cone) and, when the repulsive electrostatic forces overcome the liquid surface tension, a continuous jet erupts from the spinneret tip. Hence, solid nano- or microfibers are obtained and generally collected in a pseudo-random manner on a grounded collector (Lukáš et al., 2009). The stability of the polymer jet and the related fiber morphology depend on the spinning system properties, processing parameters and environmental conditions (Amiraliyan et al., 2009; Ismail et al., 2017; Thompson et al., 2007). In this sense, electrospinning represents an easy, versatile, and industrially scalable approach to prepare fibers with tailored properties (Kannan et al., 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).

With these premises, it is not surprising that electrospinning is nowadays widely used to prepare nanofibrous membranes for a broad range of purposes. For example, electrospun mats find several uses as biomedical and pharmaceutical devices (Cheng et al., 2018; Miguel et al., 2018), in filter materials (D. Lv et al., 2018; 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 & Bertea, 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.

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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 cyclodextrin nanofibers have gained huge interest in recent years. However, their proficient usage in 175 176 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.

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.
	MβCDs		Water, DMF, DMAc, Urea	(Celebioglu & Uyar, 2010, 2012)
	HPβCDs		Water, DMF, Urea	(Manasco et al., 2012; W. Zhang et al., 2012
	HPγCDs		Water, DMF, DMAc, Urea	(Celebioglu & Uyar, 2012)
Fundamental studies	αCDs		Water, DMF, DMAc, DMSO, water – NaOH	(Celebioglu & Uyar, 2013a)
	βCDs		Water, DMF, DMAc, DMSO, water – NaOH, ionic liquids	(Ahn et al., 2013; Celebioglu & Uyar, 2013a
	HPαCDs		Water, tap water, salty water	(Celebioglu & Uyar, 2020b)
	HPβCDs	Triclosan		(Celebioglu, Umu, et al., 2014; Celebioglu
				Uyar, 2011)
		Azobenzene		(M. Chen et al., 2013)
		Geraniol		(Aytac, Yildiz, Kayaci-Senirmak, San Keski
				Tekinay, et al., 2016)
		Camphor		(Celebioglu, Aytac, et al., 2018)
Drug and bioactive compound delivery systems	ΗΡγCDs	Triclosan		(Celebioglu, Umu, et al., 2014; Celebioglu
				Uyar, 2011)
		Geraniol		(Aytac, Yildiz, Kayaci-Senirmak, San Keski
				Tekinay, et al., 2016)
		Camphor		(Celebioglu, Aytac, et al., 2018)
-	MRCDa	Geraniol		(Aytac, Yildiz, Kayaci-Senirmak, San Keski
	MβCDs	Geranioi		Tekinay, et al., 2016)
Fast-dissolving nanofibers for the rapid		Spironolactone	EtOH	(Vigh et al., 2013)
compound administration	HPβCDs	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		(Celebioglu & Uyar, 2020a)	
acetate		(Celeblogiu & Oyar, 2020a)	
Vanillin	Water, DMF, DMAc	(Celebioglu, Kayaci-Senirmak, et al., 2016)	
Limonene		(Aytac, Yildiz, Kayaci-Senirmak, San Keskin,	
Linonene		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)
	Sulfisoxazole		(Zehra Irem Yildiz et al., 2017)
SEBβCDs	Paracetamol		(Zehra Irem Yildiz & Uyar, 2019)
	Voriconazole		(Vass et al., 2019)
	α-lipoic acid		(Celebioglu & Uyar, 2019a)
	Curcumin		(Celebioglu & Uyar, 2020c)
	Ferulic acid		(Celebioglu & Uyar, 2020e)
	Vanillin	Water, DMF, DMAc	(Celebioglu, Yildiz, et al., 2018b)
			(Aytac, Yildiz, Kayaci-Senirmak, San Keskin
	Limonene		Kusku, et al., 2016)
	Linalool		(Aytac, Yildiz, et al., 2017)
	Citral		(Aytac, Celebioglu, et al., 2018)
HPγCDs	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)
	Vitamin-A		
	acetate		(Celebioglu & Uyar, 2020a)
MOCE	Vanillin	Water, DMF, DMAc	(Celebioglu, Kayaci-Senirmak, et al., 2016)
MβCDs	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)
	γCDs		Water – DMSO	(Celebioglu & Uyar, 2013b)
Air filter materials	HPβCDs		Water – DMF	(Celebioglu, Sen, et al., 2016)
	HPγCDs		Water – DMF	(Celebioglu, Sen, et al., 2016)
		Au-NPs	Water – DMF	(Celebioglu & Uyar, 2013c)
Reducing agents for nanoparticle synthesis	HPβCDs	Ag-NPs	Water – DMF	(Celebioglu, Topuz, Yildiz, et al., 2019b)
		Pd-NPs	Water – DMF	(Celebioglu, Topuz, & Uyar, 2019a)
Sacrificial templates for ceramic hollow	HPβCDs		DMF	(Celebioglu, Vempati, et al., 2014)
nanofibers	MβCDs		DMF	(Celebioglu, Vempati, et al., 2014)
	PolyHPβCDs			(Celebioglu et al., 2017)
		Poly-benzoxazine	DFM	(Dogan et al., 2019)
				(Celebioglu, Topuz, Yildiz, et al., 2019a)
Water purification			Water – NaOH	(Celebioglu, Topuz, & Uyar, 2019b)
	Peracetylated βCDs		DMF	(Topuz et al., 2020)
Implantable coeffelds	DalvUDQCDa	Triclosan		(Hamsici et al., 2017)
Implantable scaffolds	PolyHPβCDs	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%.

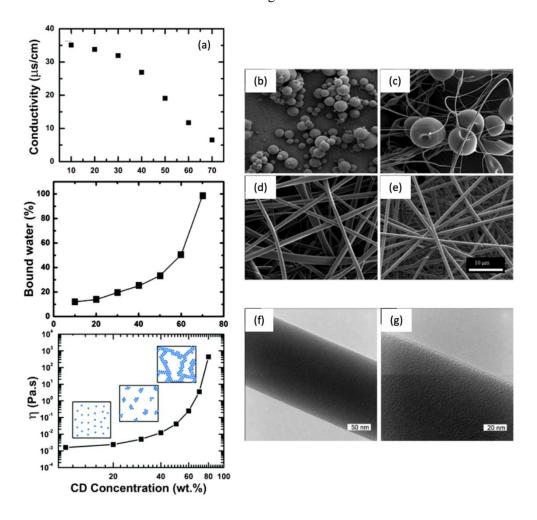




Figure 4. (a) Effect of HPβCD concentration on the conductivity, bound water and viscosity of aqueous solutions. SEM micrographs of electrospun HPβCD solutions of (b) 50 wt%, (c) 60 wt%, (d) 65 wt%, (e) and 70 wt%. TEM micrographs showing fibers electrospun from a 65 wt% solution at (f) 200000X and (g)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

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 HPBCDs 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 αCDs 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.

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219 **4.2** Applicative Studies

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

223

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

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

229

230 Encapsulation of Hydrophobic Drugs

231 Celebioglu et al. (Celebioglu, Umu, et al., 2014; Celebioglu & Uyar, 2011) first prepared HPBCD-ICs-232 triclosan and HPyCD-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 HPBCD-ICs-TCL 237 and HPYCD-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 HPBCDs 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.

244

245 Encapsulation of Volatile Bioactive Substances

Polymer-free cyclodextrin nanofibers can also be efficiently exploited to avoid the rapid evaporation of highly volatile compounds. For example, Aytac et al. (Aytac, Yildiz, Kayaci-Senirmak, San Keskin, Tekinay, et al., 2016) prepared HP β CD, HP γ CD, or M β CD geraniol inclusion complex nanofibrous mats. The authors proved the capability to encapsulate a higher amount of geraniol in pure CD nanofibers (~11 wt%) compared to polymeric membranes (~5 wt%). After both short-term temperature and long-term open-air release studies, MβCD-based nanofibers showed the slowest release of geraniol. Moreover, along with strong antibacterial activities, CD-ICs-geraniol mats also displayed efficient antioxidant properties. Yet, Celebioglu et al. (Celebioglu, Aytac, et al., 2018) prepared HPβCD-ICs- and HPγCD-ICs-camphor nanofibers from highly concentrated aqueous solutions obtaining thermally stable and manageable membranes, with HPγCD-ICs- camphor nanofibers showing a slow release of the compound.

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

Polymer-free cyclodextrin nanofibers are highly soluble in water if not subjected to a crosslinking reaction (see Section 5), but such drawback has been efficiently turned into a benefit by exploiting the fast-dissolving nature of these systems for the rapid administration of drugs and bioactive compounds in both food and pharmaceutical products (Balusamy et al., 2020). Owing to their capability to form stable inclusion complexes, cyclodextrins can indeed considerably increase the water-solubility of a wide variety of guest hydrophobic drugs and volatile molecules that can consequently be rapidly released when in contact with a 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 HPBCD 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 HPβCD 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, HPBCD-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 HPBCD 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 SBEBCD-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 HPBCD-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 HPBCDs or 301 HPyCDs and curcumin with a remarkable 100% encapsulation efficiency. Specifically, HPyCD-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 HPBCD-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 HPBCD-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 HPBCD-ICs- and HPyCD-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_βCD-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 HPBCD 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 SBEβCD-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 electrospinning of HP β CD nanofibers containing β -galactosidase, which is a protein-type drug widely used 335 336 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 HPBCD-ICs-doxycycline (DOX) intravenous 339 formulation using high-speed electrospinning with a production rate of ~ 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 HPyCDs, and MBCDs in water, DMF or DMA. The authors demonstrated that MBCDs were more stably 350 linked to vanillin leading to a higher encapsulation efficiency (up to $\sim 12 \text{ wt\%}$). 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 HPBCD-, HPyCD-, and MBCD-based nanofibrous mats encapsulating the highly volatile 356 limonene or linalool compounds. In both cases, the authors proved MBCD 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 HPBCD-, HPyCD- and MBCD-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 HPBCD-ICs- and HPyCD-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 HPBCD-ICs- and HPyCD-ICs-based electrospun 376 mats containing cineole or p-cymene. HPyCD-based nanofibers were able to encapsulate a greater amount of 377 cineole, whereas p-cymene was better preserved by the HPBCD-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 HPBCD-ICs-, HPYCD-ICs-, and MBCD-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 HPBCD- and HPyCD-based

nanofibers by Yildiz et al. (Zehra I. Yildiz et al., 2019). Bead-free and homogeneous nanofibers were obtained with considerably higher efficiency in preserving the volatile cinnamaldehyde compared to analogous PLA-based nanofibers (Y. Liu et al., 2017). Moreover, owing to their fast-dissolving nature, the prepared mats displayed strong antibacterial properties against E. Coli representing a new potential class of 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 HPBCD-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 & Uvar, 2013b) first 410 reported on the electrospinning of pristine YCDs 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 HPBCD- and HPpCD-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 HPBCD 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 HPBCD nanofibers containing in-situ synthesized silver nanoparticles (Ag-NPs) produced by

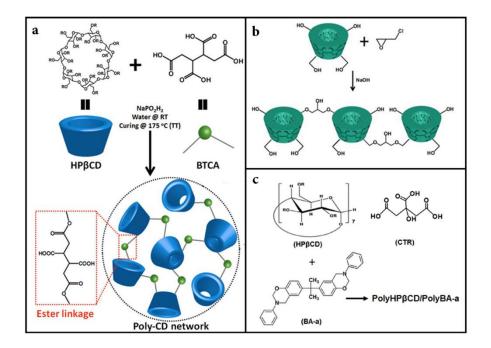
440 a single-step electrospinning process (Celebioglu, Topuz, Yildiz, et al., 2019b). The authors observed that 441 DMF solution led to thinner nanofibers compared to water, besides an increase of the mean diameter was 442 observed for both solvents by increasing the amount of loaded Ag salt precursor, 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 successfully embedded within the nanofibers, with their dimension being a function of the precursor amount 444 445 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 HPβCD/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., HPBCDs and MBCDs) 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 capability make such systems very promising for wastewater purification applications, as well as for 470 471 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.



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

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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 polyHPBCD nanofibers while maintaining their high hydrophilicity. In particular, polyHPBCD 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 polyHPBCD/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 HPBCD/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 polyHPBCD 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 polyHPβCD 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 HPBCD 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. PolyHPBCD-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. HPBCDs were subjected to crosslinking and 531 peracetylation reactions before being electrospun together with VB₂ leading to bead-free homogeneous 532 nanofibers. PolyHPBCD-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 HPβCD-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

Together with polymer-free CDs and polyCDs nanofibers, electrospun mats containing cyclodextrins have also been prepared from both native CDs and their derivatives in combination with a broad variety of carrier polymers able to provide a good spinnability and very specific properties of the mats depending on the 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).

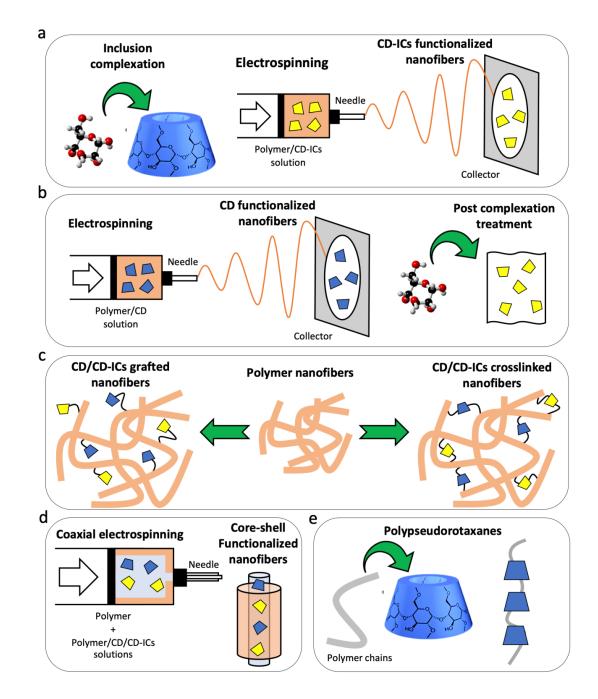


Figure 6. Schematics of preparation methods for cyclodextrin-functionalized nanofibers. (a) Electrospinning
 of polymer/CD-ICs solutions, (b) electrospinning of polymer/CDs/active compound solutions, (c) CD/CD ICs grafted or CD/CD-ICs crosslinked nanofibers, (d) coaxial electrospinning of CD/CD-ICs core
 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 polymerbased electrospun membranes functionalized with cyclodextrins will soon represent a new class of environmentally friendly materials with potential applicability in a wide range of markets. In the next two subsections, the current literature about polysaccharide- and protein-based functionalized electrospun membranes is critically discussed by highlighting the recent advances.

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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

al. (Vicini et al., 2018) used a bath collector electrospinning to obtain crosslinked composite alginate/hyaluronic acid/PEO membranes. Similarly, Ge et al. (Ge et al., 2012) and Huang et al. (X. J. Huang et al., 2007) reported on chitosan/PVA electrospun membranes for the encapsulation of bioactive compounds, whereas pullulan (PUL)/PVA nanofibers with UV-resistant properties were obtained by Qian et al. (Qian et al., 2016). Compared to pure polysaccharide nanofibrous membranes, cyclodextrin-functionalized polysaccharide nanofibers offer several advantages that will be deeply discussed in this section with a complete and up-to-date summary of the literature reported in Table 2. **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.	
			Cyclodextrin grafted nanofibers for effluent dye removal	(Norouzi et al., 2012)	
	βCDs	Cinnamon and oregano essential oils	Nanofibrous membranes with strong antibacterial properties for biomedical and food packaging applications	(Munhuweyi et al., 2018)	
Chitosan	CMβCDs	Salicylic acid	Cyclodextrin grafted nanofibers as drug delivery systems with tuneable release kinetics	(Bazhban et al., 2013)	
Cintosan	HPβCDs		Fundamental study of the effect of CDs on chitosan solution spinnability	(Burns et al., 2015; C. Xue & Wilson, 2019)	
Ро	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	(Fuenmayor et al., 2013;	
		Linionene, rerinaidenyde	stability and humidity triggered release behaviour	Mascheroni et al., 2013)	
runuan	γCDs	Eugenol	Biomedical and food packaging products with enhanced release and antioxidant properties	(Celebioglu & Uyar, 2020f	
Cellulose acetate		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)	
	t ate βCDs		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)
	αCDs		Cyclodextrin grafted nanofibers as filter materials for toluene in wastewater	(Yuan et al., 2017)
	γCDs		Cyclodextrin grafted nanofibers as filter materials for toluene in wastewater	(Yuan et al., 2017)
Hydroxypropyl cellulose	HPβCDs	Sulfisoxazole	Drug delivery systems with enhanced release abilities	(Aytac et al., 2015)
Almond gum	βCDs	Curcumin	Drug delivery systems with enhanced encapsulation capabilities for oral and gastrointestinal formulation dosages	(Rezaei & Nasirpour, 2019)
Hyaluronic acid	HPβCDs	Naproxen	Wound dressing patches with controlled drug delivery abilities enhanced water-stability	(Séon-Lutz et al., 2019)
Dandelion	βCDs	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 et al., 2012) who reported on chitosan/PVA electrospun membranes grafted with β-cyclodextrins for the 623 624 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-\beta-cyclodextrins (CMBCDs) 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 HPBCDs 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 HPBCDs with more homogeneous 632 nanofibers obtained by increasing the cyclodextrin concentration as shown in Figure 7a-d.

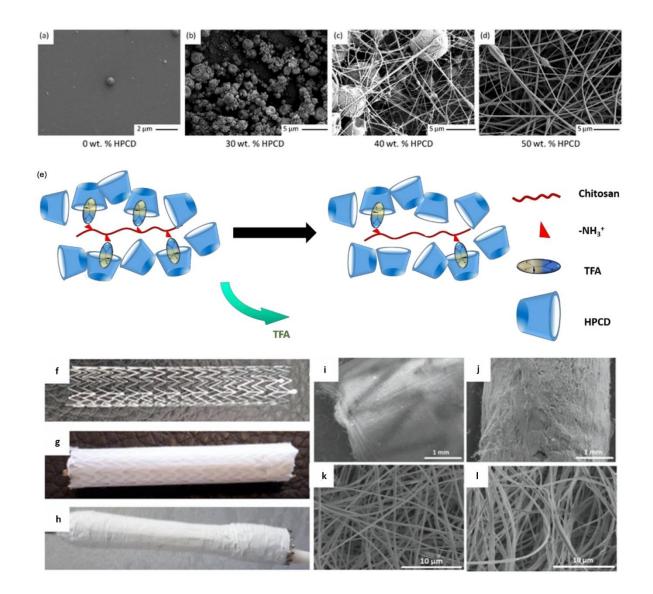


Figure 7. Fiber morphology of chitosan-based electrospun mats with an increasing HP β CD content: (a) 0 wt%, (b) 30 wt%, (c) 40 wt%, and (d) 50 wt%. Reproduced with permission from Burns et al., 2015. © The Royal Society of Chemistry 2015. (e) Schematic illustration of CH/HP β CD electrospun nanofiber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

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Such findings were attributed to the formation of a stable inclusion complex between the hydrophobic cavity of cyclodextrins and chitosan amine groups. More recently, Xue et al. (C. Xue & Wilson, 2019) carried out a solid-state spectroscopic and thermal study on CH/HP β CD electrospun nanofibers obtained using TFA as solvent. On the contrary to what reported by Burns et al. (Burns et al., 2015), it was suggested that the enhanced processability of chitosan solutions was likewise related to the formation of a ternary complex between CH, HP β CDs, and TFA with the solvent molecules acting as bridges, as schematically depicted in 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 polyHPBCDs and triclosan. Compared to pristine 651 HPBCDs, polyHPBCDs 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 HPBCD-ICs-TCL or free TCL, CH/polyHPBCD-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/BCD-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/polyHPBCD-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 HPBCD-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/βCD 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 ~93%, which was considerably higher with respect to 683 pullulan/eugenol nanofibers (i.e., ~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 BCD 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.

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

mats showed a considerably higher release most likely owing to the enhanced incorporation of SFS throughinclusion 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/βCD-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 reparation, 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 HPβCDs 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 HPBCDs 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.

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

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

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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).

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

Protein	Cyclodextrins	Encapsulated bioactive molecules	Applications	Ref.	
Soy protein isolate	βCDs	Allyl isothiocyanate	Food packaging materials with enhanced encapsulation efficiency and humidity triggered release kinetic	(Vega-Lugo & Lim, 2009)	
	αCDs		Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)	
			Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)	
	βCDs	Eucalyptus essential oil	Antibacterial food packaging products with enhanced thermal stability	(Dias Antunes et al., 2017)	
Zein	-	Quercetin	Antibacterial food packaging products	(Z. Wang et al., 2021)	
			Fundamental study of the effect of CDs on zein solution spinnability	(Kayaci & Uyar, 2012a)	
	γCDs	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		HPBCDs Tamoxifen	Transdermal drug delivery systems with prolonged-release kinetic for the	(W. Liu et al., 2016)	
SIIK HOLOIII	HPβCDs Tamoxifen		treatment of breast cancer	(w. Liu et al., 2010)	
			Water-insoluble crosslinked nanofibers as cost-effective water pollutant	(Yu Chen et al., 2018)	
			removal	(1 u Chen et al., 2018)	
Gelatin	βCDs –	βCDs	Respiratory filter media for filtration of aerosols and volatile organic	(Kadam et al., 2021)	
			compounds		
	-	Galangal root oil	Active food packaging products with antibacterial properties	(Zhou et al., 2020)	
	HPβCDs	Ciprofloxacin	Fast-dissolving drug delivery systems for oral formulation dosages	(Aytac et al., 2019)	
Gliadin	HPβCDs	HPBCDs Ferulic acid	Biomedical and food products with enhanced thermal stability and fast release	(Sharif et al., 2018)	
Gliadin	nrpcDs	refulic aciu	properties	(Shann et al., 2018)	
Hordein	βCDs	N,N'-methylene bisacrylamide	Recyclable filter materials for the efficient removal of heavy metals from	(Guan et al., 2019)	
moruem	pcDs		wastewater	(Ouali et al., 2019)	

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

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/BCD-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 BCD-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 HPBCD-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.

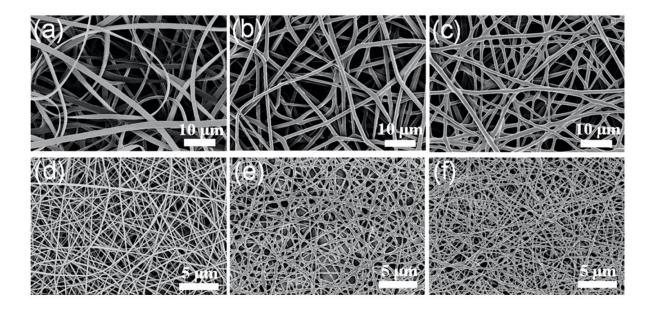


Figure 8. SEM micrographs of (a) as-spun RSF nanofibers, (b) RSF nanofibers after ethanol spraying
treatment, (c) RSF nanofibers after acetone immersion treatment, (d) as-spun RSF/HPβCD-ICs-TAM
nanofibers, (e) RSF/HPβCD-ICs-TAM nanofibers after ethanol spraying treatment, (f) RSF/HPβCDs-ICsTAM nanofibers after acetone immersion treatment. Reproduced with permission from W. Liu et al., 2016.
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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 HPBCD and ciprofloxacin (CIP) complexes as fast-843 dissolving drug delivery systems. The authors were able to prove that the bead-free nanofibers encapsulating 844 HPBCD-ICs-CIP showed to be more rapidly dissolved compared to gelatin/CIP nanofibers, thus pointing out 845 the capability of HPBCDs 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/BCD-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.

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

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

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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	СА	Cellulose acetate
936	CTR	Citric acid
937	CD-ICs	Cyclodextrin inclusion complexes
938	CDs	Cyclodextrins
939	Ce	Entanglement concentration
940	СН	Chitosan
941	CIP	Ciprofloxacin
942	CMβCDs	Carboxymethyl-
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	НА	Hyaluronic acid
971	HD	Hordein
972	HPC	Hydroxypropyl cellulose

973	HPαCDs	2-hydroxypropyl-aCDs
974	HPβCDs	2-hydroxypropyl-βCDs
975	HPγCDs	2-hydroxypropyl-γCDs
976	HSES	High speed electrospinning
977	IKAV	Adamantane-6-aminohexanoic acid-GGKIKVAV-Am
978	KK	Adamantane-6-aminohexanoic acid-GGKK-Am
979	L. monocytogenes	Listeria monocytogenes
980	LCEO	Litsea cubeba essential oil
981	MB	Methylene blue
982	MBA	N,N'-methylene bisacrylamide
983	МО	Methyl orange
984	MβCDs	Randomly methylated-BCDs
985	NaOH	Sodium hydroxide
986	NAP	Naproxen
987	Ni	Nickel
907	INI	Nickei
988	NMR	Nuclear magnetic resonance spectroscopy
988	NMR	Nuclear magnetic resonance spectroscopy
988 989	NMR NPs	Nuclear magnetic resonance spectroscopy Nanoparticles
988 989 990	NMR NPs PAA	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid)
988 989 990 991	NMR NPs PAA PAN	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile
988 989 990 991 992	NMR NPs PAA PAN PAP	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol
988 989 990 991 992 993	NMR NPs PAA PAN PAP PCL	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol Polycaprolactone
988 989 990 991 992 993 994	NMR NPs PAA PAN PAP PCL Pd	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol Polycaprolactone Palladium
988 989 990 991 992 993 994 995	NMR NPs PAA PAN PAP PCL Pd Pd-NPs	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol Polycaprolactone Palladium
988 989 990 991 992 993 994 995 996	NMR NPs PAA PAN PAP PCL Pd Pd-NPs PEO	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol Polycaprolactone Palladium Palladium nanoparticles
988 989 990 991 992 993 994 995 995 996 997	NMR NPs PAA PAN PAP PCL Pd NPs PEO PET	Nuclear magnetic resonance spectroscopy Nanoparticles Poly(acrylic acid) Polyacrylonitrile p-aminophenol Polycaprolactone Palladium Palladium nanoparticles Poly(ethylene oxide)

1002PolyBA-aPoly benzoxazine1003PolyCDsCyclodextrin-based polymers1004PolyHPβCDsPoly-2-hydroxypropyl-βCDs1005PSPolystyrene1006PULPullulan1007PVAPolyvinyl alcohol1008PVPPolyvinylpyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SFSulfisoxazole1018SHPSoy protein isolate1019SVSinwastatin1020TAMTamoxifen1021TAMTimoxifen	1001	PNP	p-nitrophenol
1004PolyHPβCDsPoly-2-hydroxypropyl-βCDs1005PSPolystyrene1006PULPullulan1007PVAPolyvinyl alcohol1008PVPPolyvinylpyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SFSSulfsoxazole1018SHPSoy protein isolate1019SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen	1002	PolyBA-a	Poly benzoxazine
1005PSPolystyrene1006PULPullulan1007PVAPolyvinyl alcohol1008PVPPolyvinylpyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSulfisoxazole1017SFSSulfisoxazole1018SHPSoy protein isolate1020SVSimwastatin1021JBGSwim bladder gelatin1023TBZThiabendazole	1003	PolyCDs	Cyclodextrin-based polymers
1006PULPullulan1007PVAPolyvinyl alcohol1008PVPPolyvinyl pyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSodium hypophosphite1018SHPSoy protein isolate1020SVSimvastatin1021TAMTamoxifen1023TBZThiabendazole	1004	PolyHPβCDs	Poly-2-hydroxypropyl-βCDs
1007PVAPolyvinyl alcohol1008PVPPolyvinylpyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SFSilk fibroin1016SFSulfisoxazole1017SFSSodium hypophosphite1018SNSoy protein isolate1020SVSimvastatin1021TAMTamoxifen1023TBZThiabendazole	1005	PS	Polystyrene
1008PVPPolyvinylpyrrolidone1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodjum hypophosphite1019SVSinvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1006	PUL	Pullulan
1009RCRegenerated cellulose1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodjum hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1007	PVA	Polyvinyl alcohol
1010RHRelative humidity1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfosxazole1018SHPSodjum hypophosphite1019SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1008	PVP	Polyvinylpyrrolidone
1011RSFRegenerated silk fibroin1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISimvastatin1020SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1009	RC	Regenerated cellulose
1012S. AureusStaphylococcus Aureus1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodjum hypophosphite1019SPISimvastatin1020SVSimvastatin1021TAMTamoxifen1023TBZThiabendazole	1010	RH	Relative humidity
1013SASodium alginate1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1011	RSF	Regenerated silk fibroin
1014SEBβCDsSulfobutylether-β-cyclodextrins1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1012	S. Aureus	Staphylococcus Aureus
1015SEMScanning electron microscopy1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1023TBZThiabendazole	1013	SA	Sodium alginate
1016SFSilk fibroin1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1014	SEBβCDs	Sulfobutylether-\beta-cyclodextrins
1017SFSSulfisoxazole1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1015	SEM	Scanning electron microscopy
1018SHPSodium hypophosphite1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1016	SF	Silk fibroin
1019SPISoy protein isolate1020SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1017	SFS	Sulfisoxazole
1020SVSimvastatin1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1018	SHP	Sodium hypophosphite
1021SBGSwim bladder gelatin1022TAMTamoxifen1023TBZThiabendazole	1019	SPI	Soy protein isolate
1022TAMTamoxifen1023TBZThiabendazole	1020	SV	Simvastatin
1023 TBZ Thiabendazole	1021	SBG	Swim bladder gelatin
	1022	ТАМ	Tamoxifen
1024 TCL Triclosan	1023	TBZ	Thiabendazole
	1024	TCL	Triclosan
1025 TEM Transmission electron microscopy	1025	TEM	Transmission electron microscopy
1026 TF Tetrafluoroterephthalonitrile	1026	TF	Tetrafluoroterephthalonitrile
1027 TFA Trifluoroacetic acid	1027	TFA	Trifluoroacetic acid
1028TGAThermogravimetric analysis	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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