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Review

Electrochemical detection of bisphenols in food: A review

Yuhuan Zhang [a,b,1], Yanan Lei [a,b,1], Hao Lu [a,b], Lin Shi [a,b], Peng Wang [a,b], Zeshan Ali [a,b], Jianke Li [a,b,*]

[a] College of Food Engineering and Nutritional Science, Shaanxi Normal University, Xi'an 710119, China
[b] University Key Laboratory of Food Processing Byproducts for Advanced Development and High Value Utilization, Xi'an 710119, China

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ABSTRACT

Bisphenols (BPs) are worldwide used organic compounds in plastics, belonging to the group of endocrine disrupting chemicals (EDCs) which exhibits endocrine disruption to beings. Migration of BPs from food contact materials like plastic containers, epoxy coatings in metal cans and thermal papers, would results in bioaccumulation of BPs in human beings, causing adverse health effects. Therefore, sensitive and selective determination of BPs in food is needed. Among different strategies have been explored for the detection of BPs, electrochemical sensors with relatively high sensitivity and fast response are promising. This paper is devoted to comprehensively review the developed electrochemical methods for BPs sensing in food, so that to find a direction for developing low cost, high accuracy and compatibility sensors toward the sensitive and selective detection of BPs. Different electrochemical technologies categorized by recognition agents, aptamers, enzymes, molecularly imprinted polymers and nanomaterials are discussed and summarized in their mechanisms, usages, merits and limitations. The challenges and further perspectives in the development of electrochemical sensors is also discussed.

1. Introduction

Endocrine disrupting chemicals (EDCs) are exogenous substances that interfere with the endocrine system, blocking the metabolism of steroid hormones and causing various disorders related to diabetes, obesity, cardiovascular diseases, carcinogenicity and neurotoxicity (Sofen & Furst, 2020). Bisphenols (BPs), containing two hydroxyphenyl groups, are popular members of EDCs. BPs, especially the bisphenol A (BPA), possess potent endocrine disrupting activities and are widely applied as component of plastics and resins used in food packaging materials, bottle tops, tableware, water pipes and epoxy coatings in metal cans (Dhanjai et al., 2018; Yin et al., 2011). Migration of BPs from food contact materials would result in the exposure of BPs to human bodies, raising concerns for food safety. It should be noted that BPA as the most common used BPs has been detected in many food samples (drinking water (Razavipanah et al., 2019), milk (Ensafi, Amini, & Rezaei, 2018), canned food et al. (El Moussawi et al., 2019) in the range of 14.0 to 521.0 ng·ml^{-1}.

A lot of studies have been focused on effects of BPs on human health, of which BPA is identified with reproductive damage, neural disorders and behavioral dysfunction (Chen et al., 2017). U.S. Food and Drug Administration (FDA) has banned the usage of BPA in baby bottles and sippy cups. In 2016, The European Union issued regulations to reduce the specific migration limit of food exposure to BPA from 0.6 mg·kg^{-1} to 0.05 mg·kg^{-1}. In Japan, the dissolution limit of BPA in polycarbonate food containers is set as 2.5 mg·kg^{-1} (Peyre et al., 2014).

After onset of the strict regulations on production and usage of BPA, bisphenol B (BPB) (Sui et al., 2012), bisphenol S (BPS) (Peng, Wang, & Wu, 2019), bisphenol AF (BPAF) (Yang et al., 2014) and bisphenol F (BPF) (Wang et al., 2014) have emerged as alternatives of BPA. Their chemical structures are shown in Fig. 1. Otherwise, those BPA analogues are proved to have same even stronger estrogenic effect than BPA due to their similar chemical structure with BPA (Liang et al., 2020; Thoene et al., 2020). Researches have shown that exposure to BPA analogues may result in adverse effects on reproduction (Kim et al., 2019; Thoene et al., 2020), neurology (Santoro et al., 2019), oxidative stress (Liu et al., 2017; Thoene et al., 2020), metaboly and bone (Chin, Pang, & Mark-Lee, 2018). In food samples, BPs have been detected in concentrations of 1.6 to 26.2 ng·ml^{-1} for BPF and 16.0 to 67.0 ng·ml^{-1} for BPB (Dhanjai et al., 2018; Grumetto et al., 2013). Therefore, it has become an urgent issue to

* Corresponding authors at: College of Food Engineering and Nutritional Science, Shaanxi Normal University, Xi'an 710119, China.
E-mail address: jiankel@snnu.edu.cn (J. Li).
[1] These authors are contributed equally.

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establish a reliable analytical method for the detection of BPs in food samples.

To date, the major analytical approaches for the detection of BPs are based on chromatographic or hyphenated methods, such as high performance liquid chromatography (HPLC) (Watabe et al., 2004), high performance liquid chromatography-tandem mass spectrometry (HPLC-MS) (Alnaimat, Barciela-Alonso, & Bermejo-Barrera, 2019), gas chromatography (GC) (Sun et al., 2016), gas chromatography-mass spectrometry (GC–MS) (Cunha et al., 2020) and so on. These techniques have high sensitivity and good stability to BPs, yet are limited to facile in situ detection due to the used large instruments as well as tedious pretreatment procedures. Some rapid sensing strategies like spectrophotometry (Dhanjai et al., 2018), immunoassay (Sheng et al., 2018), capillary electrophoresis (Gugoasa, 2019), fluorimetry (Varmira, Saed-Mocheshi, & Jalalvand, 2017), and electrochemical detection methods have thus caught great attention in recent years. Among them, electrochemical sensors possessing low-cost, rapidity, extremely high-sensitivity, simplicity, ease of miniaturization and in situ analysis is very promising (Karimian et al., 2019; Khanmohammadi et al., 2020; Ragavan, Rastogi, & Thakur, 2013). A sensor is a device which provides a usable output in response to a specified measurand. The electrochemical sensors can convert the BPs concentrations into electrochemical signals. A summary about the electrochemical detection of BPs in food will help to deeply understand the detection mechanism and development trend of BPs sensing, and thus to find a clear direction for low cost, high accuracy and compatibility BPs sensors. This paper present and evaluate recent electrochemical works on the detection of BPs in food, aiming to guide the detection of BPs, and also provide a reference for the development and advancement in the relevant fields of research, like the monitoring of pesticides and antibiotics.

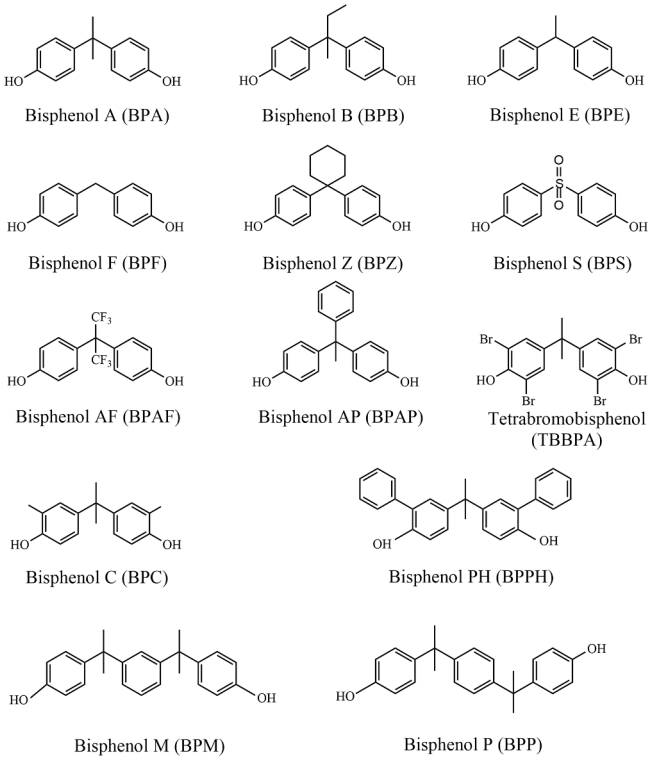
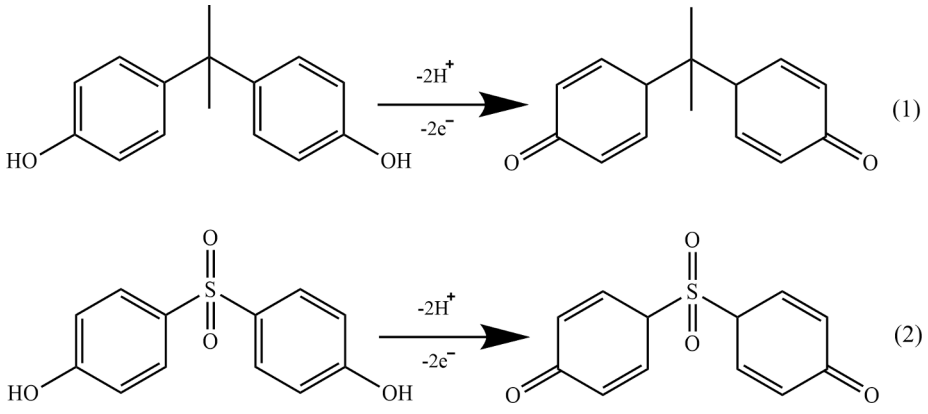


Fig. 1. Chemical structures of BPs.

2. Electrochemical detection of BPs

BPs are electrochemically-active molecules with two phenolic groups that can be directly electrooxidized to corresponding o-quinine at positive potentials. The oxidation of BPs is an irreversible two-electron and two-proton process (Li et al., 2012), which can be illustrated as eq. (1) for BPA and (2) for BPS:



Although all the BPs have similar electrooxidation mechanism, their voltammograms are very different, for example the oxidation peak of BPA is at potential of 0.4 V–0.7 V (vs. Ag/AgCl), while the BPS is at a more positive potential of 0.7 V–1.0 V (Pang et al., 2020; Rao et al., 2018; Wang et al., 2014; Zhu et al., 2016), making the identification of various BPs much easier.

However, the direct determination of BPs is still difficult due to the weak response of BPs in conventional electrochemical sensors (Ragavan, Rastogi, & Thakur, 2013). Researchers have developed diverse types of electrode materials to increase the effective electrode surface area or electron transfer kinetics between the electrode surface and the redox centers of analytes (Zhang et al., 2020). In real food samples, a wide range of interference species may exist, hence recognition agents with selective BPs recognition capability are necessary. In this review, the developed electrochemical sensors have been categorized according to their recognition elements such as aptamers, enzymes, molecularly imprinted polymers and nanomaterials with selective catalytic abilities for BPs.

2.1. Electrochemical aptasensors

Aptamers are selected oligonucleotides that can specifically bind to target molecules from specific oligonucleotide libraries by the exponential enrichment of ligand evolution technique (Razavipanah et al.,

Table 1
Different electrochemical sensors for BPs

Electrochemical sensors	BPs	LOD/$_{\text{Linear range}}$ (nM)	Recovery %	Sample	Ref.
Electrochemical aptasensors:High specificity and selectivity. Relatively poor stability, repeatability and reproducibility.					
MCH[1]/aptamer/Au/$CuFe_2O_4$-Pr-SH[2]/MWCNTs[3]/GCE.	BPA	$0.025/_{0.05-9}$	98.9 –110.5	Water Milk Juice	(Baghayeri et al., 2018)
MWCNT/SiO_2@Au/aptamer/GCE[4]	BPA	$0.01/_{0.1-100}$	96.0 – 108.0	Milk Orange juice	(Razavipanah et al., 2019)
Aptamer/ NP-Au[5]/GCE	BPA	$4.4\times10^{-5}/(_{0.11-8.76})^{-3}_{\times10}$	95.0 – 103.0	Red wine	(Shi et al., 2018)
MB[6]-Aptamer/Au/MWCNTs/CT[7]/Au/PET[8]	BPA	$8\times10^{-6}/_{10-1}^{-5}$	88.0 – 106.2	Bottles Water Milk	(Li et al., 2019a, 2019b)
aptamer/PCB[9]-based/GCE	BPA	$1.52\times10^{-7}/_{10-0.01}^{-6}$	75.1 – 106.1	Canned foods	(Mirzajani et al., 2017)
DNA/MCH/BPA-aptamer-ssDNA[10]-B/PBIB[11]/FMMA[12]/ gold electrode	BPA	$5.9\times10^{-8}/_{10-100}^{-5}$	95.23 – 98.4	Water	(Li et al., 2019a, 2019b)
Enzyme-based sensors:High catalytic activity and selectivity. Relatively poor stability, repeatability and reproducibility.					
Tyr[13]-MWCNTs/GCE	BPA	$500/_{2000-10}^{5}$	94.3 – 105.5	Plastic bottles	(Ren et al., 2011)
rGO-Fe_3O_4NPs[14]/Chit95[15]/TvL[16]/GCE	BPA	$18.1/_{0.025-25}$	107.0 –124.0	Bottled water	(Fernandes, Campina, & Silva, 2020)
CuMOFs[17]-Tyr-Chit[18]/GCE	BPA	$13/_{50-3000}$	–	Water	(Lu et al., 2016)
Tyr-AuNPs[19]/SPCE[20]	BPA	$10/_{42-3.5\times10}^{4}$	–	Water	(Alkasir et al., 2010)
Tyr-Fe_3O_4/SPCE	BPA	$8.3/_{22-4\times10}^{4}$			
Tyr-APS[21]/Au-GN[22]/GCE	BPA	$7.88/_{44-4.4\times10}^{4}$	87.4 – 110.7	Olive oilChips	(Wu et al., 2019)
Tyr-NiNPs/SPCE	BPA	$7.1/_{91-4.8\times10}^{4}$	–	Water	(Alkasir et al., 2010)
Tyr/CFP[23]	BPA	$5/_{10-1000}$	–	Tap water	(Yuan et al., 2011)
BCNP[24]/Tyr/Nafion/GCE	BPA	$3.18/_{20-10}^{4}$	96.7 – 108.6	Water	(Liu et al., 2019a)
XOD[25]/GCE	BPA	$1/_{1-41}$	97.9 – 103.0	Mineral water	(Ben Messaoud et al., 2018)
CuMOFs-Tyr-Chit/GCECuMOFs-Tyr-Chit/GCE	BPF	$16/_{50-3000}$	–	Water	(Lu et al., 2016)
	BPE	$15/_{50-3000}$			
Molecularly imprinted sensors:High stability, repeatability and selectivity. Poor conductivity and catalytic activity.					
MIP[26]/GCE	BPA	$40/_{100-5\times10}^{4}$	93.7 – 94.5	Tap water	(Tan et al., 2016)
MWCNT-MIP/GCE	BPA	$30/_{200-4.5\times10}^{4}$	92.7 – 96.0	Tap water	(Liu et al., 2019b)
MIPN[27]/GCE	BPA	$8/_{20-1000}$	71.2 – 97.3	Tap water Drinking water	(Ali, Mukhopadhyay, & Jana, 2019)
GO[28]/APTES[29]–MIP/GCE	BPA	$3/_{6-2\times10}^{4}$	96.8 – 106.2	Mineralised water Milk	(Dadkhah et al., 2016)
MIPs/AuNPs/GCE	BPA	$1.1/_{15-5.5\times10}^{4}$	97.6 – 106.5	Milk	(Zhao et al., 2017)
MIP/GCE	BPA	$0.02/_{0.1-4\times10}^{5}$	–	Baby feeding bottles	(Anirudhan, Athira, & Sekhar, 2018
GR[30]/MIPs/ABPE[31]	BPA	$4.2\times10^{-4}/_{1.41\times10-1.23\times10^{6}}^{-3}$	95.4 – 102.6	Packaging materials Water	(Xu et al., 2016)
sMIP[32]/GCE	BPAF	$60/_{100-10}^{4}$	93.0 – 103.0	Water	(Zhang et al., 2017)
hNiNS[33]/GQDs[34]/MIPs/GCE	BPS	$30/_{100-5\times10}^{4}$	94.4 – 99.6	Plastic bottles	(Rao et al., 2018)
Nanomaterials-based sensors:Strong catalytic activity. High stability, repeatability and reproducibility. Poor selectivity.					
GR[35]/GCE	BPA	$46/_{50-1000}$	90	Plastic bottles	(Ntsendwana et al., 2012)
NiNPs/NCN[36]/CS/GCE	BPA	$45/_{100-1.5\times10}^{4}$	96 – 105	Milk	(Wang, Yin, & Zhuang, 2020)
Cu-MOFs/ERGO[37]/GCE	BPA	$6.7/_{20-9\times10}^{4}$	98.5 – 105.4	Plastic products	(Li et al., 2018)
Lac[38]/Ag-ZnONPs/MWCNTs/C-SPE[39]	BPA	$6/_{500-2990}$	89.3 – 109.3	Plastic bottles	(Kunene et al., 2018)
MoS_2-Chi[40]-AuNPs/GCE	BPA	$5/_{50-10}^{5}$	94.2 – 105.2	FoodWater	(Huang et al., 2014)
MWCNTs-COOH/GCE	BPA	$5/_{10-10}^{4}$	98.4 – 102.8	Food packaging	(Li et al., 2010)
$CoFe_2O_4$/GCE	BPA	$3.6/_{500-10}^{4}$	95.5 – 102.0	Milk Tap water	(Liu et al., 2020)
Mg-Al-SDS[41] LDH[42]/GCE	BPA	$2/_{8-2800}$	96.4 – 102.6	Tap water	(Yin et al., 2011)
Arg-GR[43]/GCE	BPA	$1.1/_{5-40}$	98.9 – 102.2	Plastic products	(Zhang et al., 2012)
GR/ABPE	BPA	$0.6/_{0.8-10}$	97.6 – 103.8	Infant nursing bottlesWater bottles	(Deng, Xu, & Kuang, 2013)
rGO-rC$_{60}$[44]/GCE	BPS	$500/_{10-10}^{3\ 5}$	84.2 – 91.7	Milk	(Zhu et al., 2016)
ILs@HPS-Ni/$CdFe_2O_4$[45]/GCE	BPS	$5.37/_{8-1.6\times10}^{4}$	91.5 – 106.9	Plastic bottles	(Wang et al., 2017)
ILs@HPS-Ni/$CdFe_2O_4$/GCE	BPAP	$4.99/_{6.9-1.379\times10}^{4}$	–	Plastic bottles	(Wang et al., 2017)
MWCNTs-COOH[46]/GCE	BPAF	$1.7\times10^{5}/_{(0.60-1.6)\times10}^{6}$	97.5 – 102.5	Water	(Yang et al., 2014)
MWCNTs-COOH/GCE	BPF	$0.549/_{0.592-30}$	93.8 – 105.3	Water Milk	(Wang et al., 2014)

[1]MCH: 6-mercapto-1-hexanol; [2]$CuFe_2O_4$-Pr-SH: functional cupper magnetic nanoparticles; [3]MWCNTs: multi-walled carbon nanotubes; [4]GCE: glassy carbon electrode; [5]NP-Au: nanoporous gold; [6]MB: methylene blue; [7]CT: carbon tape; [8]PET: polyethylene terephthalate electrode; [9]PCB: printed circuit board; [10]ssDNA: single-stranded DNA; [11]PBIB: propargyl-2-bromoisobutyrate; [12]FMMA: ferrocene methyl methacrylate; [13]Tyr: tyrosinase; [14]rGO-Fe_3O_4 NPs: reduced graphene oxide/ferrous-ferric oxide nanoparticles; [15]Chit95: chitosan DD = 95%; [16]TvL: Trametes versicolor; [17]MOFs: metal – organic frameworks; [18]Chit: chitosan; [19]AuNPs: gold nanoparticles; [20]SPCE: screen-printed graphite carbon electrode; [21]APS: Au-Pt@SiO_2; [22]Au-GN: Au-graphene; [23]CFP: carbon fiber paper; [24]BCNP: biochar nanoparticle; [25]XOD: xanthine oxidase; [26]MIP: molecularly imprinted polymers; [27]MIPN: molecularly imprinted polymer nanocomposite; [28]GO: graphene oxide; [29]APTES: 3-aminopropyltriethoxysilane; [30]GR: graphene; [31]ABPE: acetylene black paste electrode; [32]sMIP: soluble molecularly imprinted polymers; [33]hNiNS: hollow nickel nanospheres; [34]GQDs: graphene quantum dots; [35]GR: graphene; [36]NiNPs/NCN: nickel nanoparticles/nitrogen-doped carbon nanosheet; [37]ERGO: electrochemically reduced graphene oxide; [38]Lac: laccase; [39]C-SPE: carbon-screen printed electrodes; [40]Chi: chitosan; [41]SDS: sodium dodecyl sulfate; [42]LDH: layered double hydroxide; [43]Arg-GR: arginine functionalized graphene; [44]rGO-rC60: co-reduced graphene oxide-C60 nanocomposite; [45]ILs@HPS-Ni/$CdFe_2O_4$: Ionic liquids@hollow porous spherical Ni-loaded $CdFe_2O_4$; [46]MWCNTs-COOH: carboxyl functionalized multi-walled carbon nanotubes.

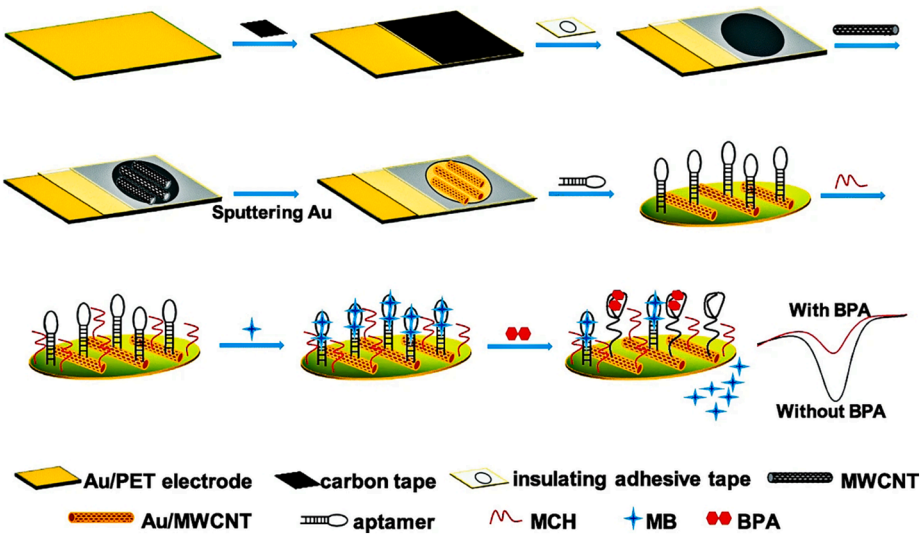


Fig. 2. Schematic representation of the aptasensor (Li et al., 2019a, 2019b).

2019). Aptamers have similar properties to antibodies and are superior to them, they have stronger specificity to targets, and can distinguish the fine structure of target molecules which means they only bind specifically to target molecules but their structural analogues (Mirzajani et al., 2017). Aptamers can be synthesized in large quantities in vitro, and have the characteristics of simple preparation, easy acquisition, low cost and easy storage (Yu et al., 2019). With the combination of aptamers and sensing technologies, different electrochemical aptasensors are developed to achieve accurate detection of BPs molecules, as shown in Table 1.

Haiyu Li et al. (Li et al., 2019a) designed an electrochemical aptasensor for the detection of BPA using methylene blue (MB) and gold-coated multiwalled carbon nanotubes, that is (Au/MWCNTs)-MB-aptamer, to realize double signal amplification (Fig. 2). When BPA was present, the competitive binding of BPA and MB to aptamer could cause the conformational change of MB-aptamer and the release of MB, the electrochemical signal from MB reduction would decreases correspondingly. Under optimal experimental conditions, the current drops linearly with the logarithm of BPA concentrations over a range from 10 fM to 1 nM, the limit of detection (LOD) is 8 fM and the recoveries of spiked BPA is 88.0–106.2%, revealing the applicability of this method in detection of real food samples. Manman Li et al. (Li et al., 2019b) constructed a novel BPA aptasensor based on an electrochemically mediated atom transfer radical polymerization (eATRP) signal amplification. The hairpin DNA with 5′-end modified with sulfhydryl group and 3′ -end modified with azide group were immobilized on a gold electrode through Au-S bond. A double-stranded DNA consists of BPA-binding aptamer is added to open the hairpin structure, making the azide groups at the 3′ end be exposed. A propargyl-2-bromoisobutyrate was used as an initiator of eATRP, and linked to the 3′ -end azide group by click chemistry reaction. The eATRP then occurred for polymerization of ferrocene methyl methacrylate. Benefit by the unique signal amplification design, this aptasensor shows a low LOD of 59 aM as well as a good selectivity versus 100-fold structural analogs. A high recovery of 95.23–98.40% is obtained by this aptasensor in the detection of BPA spiked food samples.

Electrochemical aptasensors have also shown reliably quantification ability for BPA detection in unspiked samples. Baghayeri's group (Baghayeri et al., 2018) developed a label-free BPA aptasensor using gold nanoparticles (AuNPs), cupper magnetic nanoparticles and multiwall carbon nanotubes (MWCNTs), with which the BPs in real samples (mineral water, milk and juice) were directly detected with or without simple preliminary treatments, indicating a valuable application in food safety. Similarly, Rezaei's group (Ensafi, Amini, & Rezaei, 2018) reported a molecularly imprinted electrochemical aptasensor that was applied in the quantitation of BPA in spiked and unspiked food samples including water, milk and milk powder.

Aptasensors have high specificity to BPs and can easily realize signal amplification, making the sensitive and selective determination of BPA in real food samples possible. But in most of the electrochemical BPs aptasensors, modification of aptamers is needed, which is complicated, time-consuming and relatively expensive. On the other hand, the as-used aptamers are easily inactivated to temperature, pH or self-inhibition, making the detection weak stability and repeatability. Further, not every BPs has its binding aptamer, until now, only the BPA and BPS have been monitored using aptasensors (Zhang, Yao, Wang, Liang, & Qin, 2018).

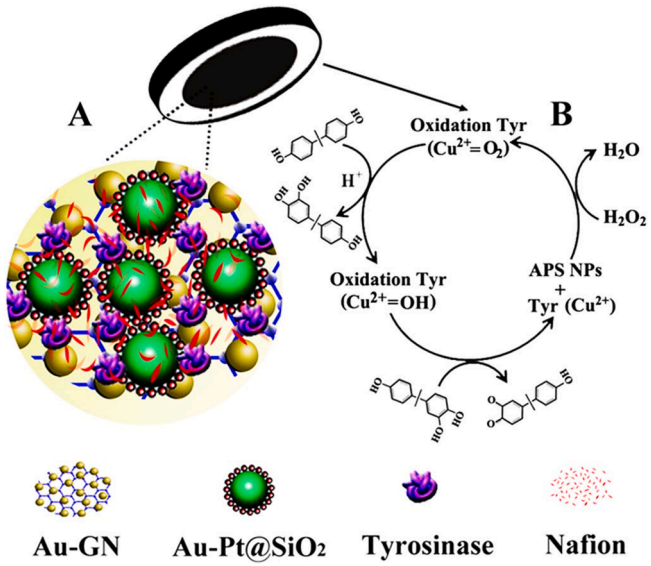
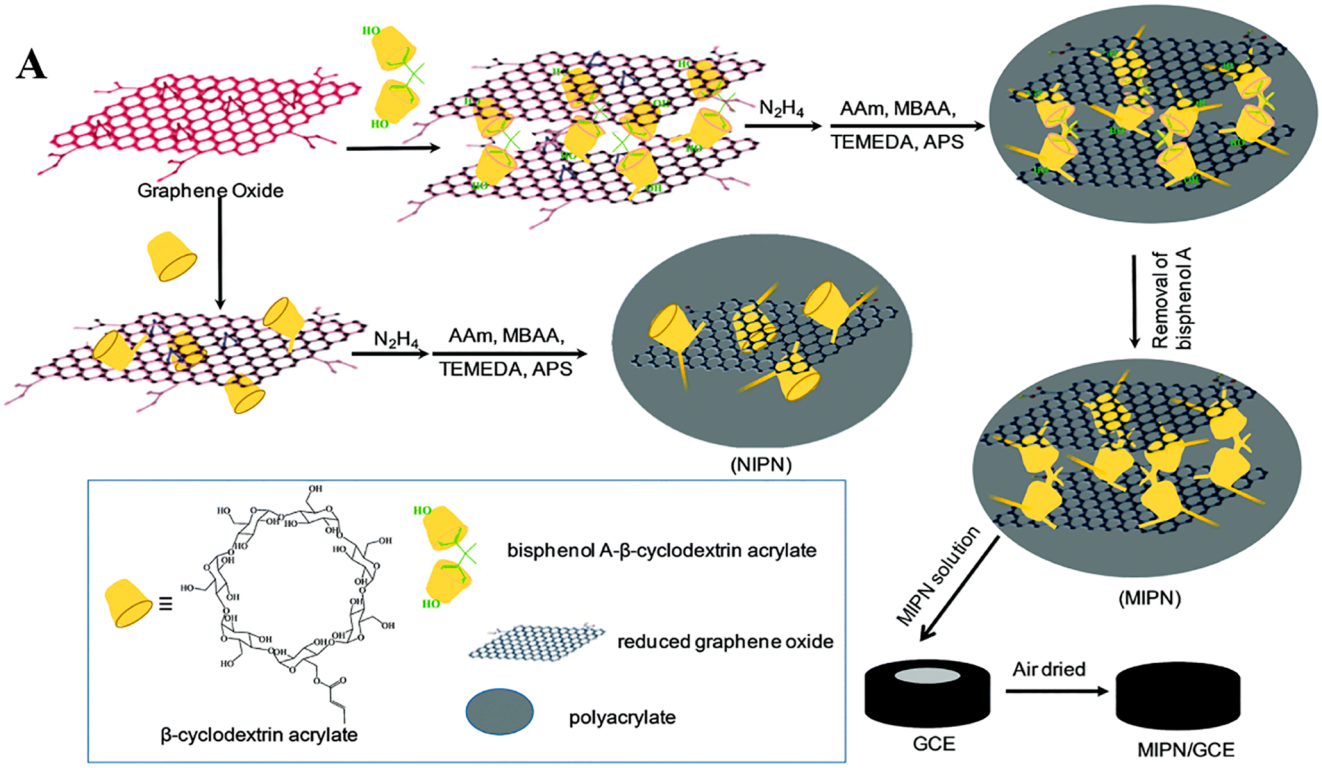


Fig. 3. (A) Illustration for the construction of tyrosinase (Try) biosensor based on Try/APS/Au-GN. (B) Possible oxidation process of BPA on the developed electrochemical sensor. (C) differential pulse voltammetry (DPV) of the nafion/Tyr/APS/Au-GN/GCE in PBS (pH = 6.0, 0.1 M KCl) with different BPA concentrations from down to up: 0.01, 0.1, 0.3, 0.5, 1.0, 3.0, 5.0, 8.0 mg·L^{-1}. (D) Linear calibration plot of DPV response to different concentrations of BPA (Wu et al., 2019).

2.2. Enzyme-based electrochemical sensors

Biorecognition elements like enzymes and receptors have been used to improve the performance of sensors (Karimian et al., 2020; Mahmoudi et al., 2019a). Enzymes have intrinsic selectivity and high catalytic activity for target molecules and are extensively used for the detection of BPs (Dhanjai et al., 2018). Tyrosinase is the most common used enzyme in BPs determination, comparing with the other oxidation–reduction enzyme like superoxide dismutase (SOD) and horseradish peroxidase (HRP). Tyrosinase is an *ortho*-hydroxylation oxidase that possesses catalytic activity for o-diphenol and monophenol (Lu et al., 2016). As shown in Table 1, different enzyme-based electrochemical BPs sensors are listed.

Xianbo Lu et al. developed a MOFs-based tyrosinase nanosensor to clearly investigate the electrochemical behavior and detection mechanisms of nine commonly used BPs (Lu et al., 2016). This nanosensor shows a selective response to bisphenol Z (BPZ), BPB, BPF, bisphenol E (BPE) and BPA, but no response to BPS, bisphenol AP (BPAP), BPAF etal., implying the electrochemical detection performance is affected not only by the BPs molecular structure, but also by the properties of substituent group on BPs framework. Xie's group developed a sensitive BPA sensor based on tyrosinase/Au-Pt@SiO_2/Au-graphene (Try/APS/Au-GN) (Wu et al., 2019). The reaction mechanism is proposed in Fig. 3. A high electrochemical performance of this biosensor is achieved with a



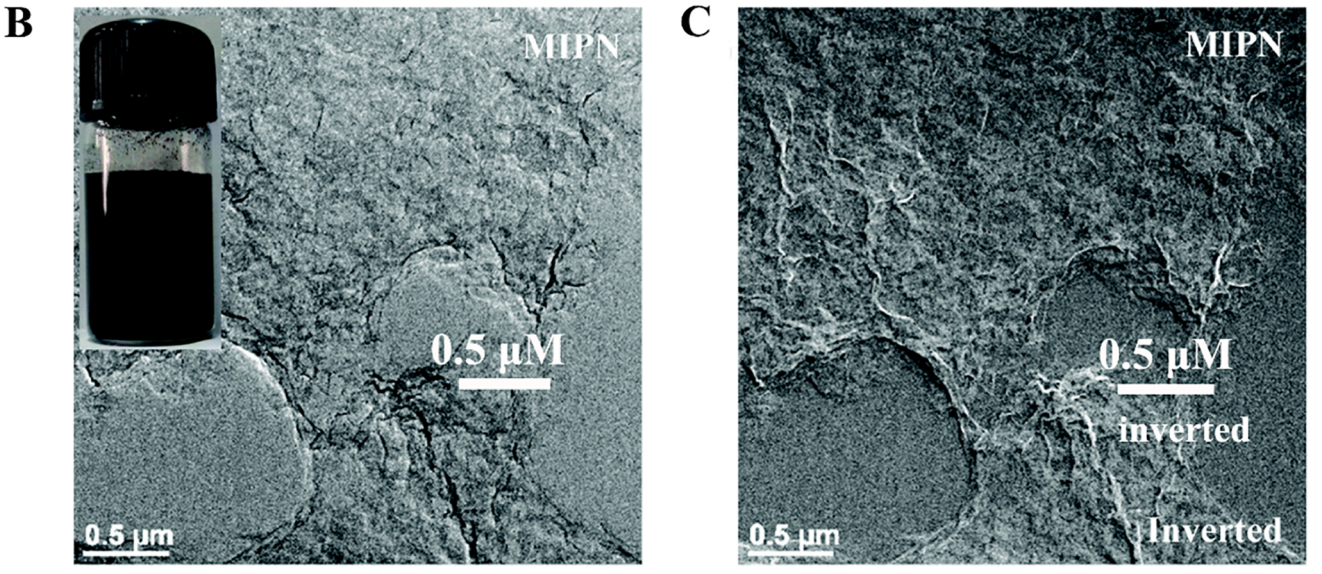


Fig. 4. (A) Representation for the synthesis of a MIPN for BPA and a non-imprinted nanocomposite (NIPN) and modification of electrodes for electrochemical measurement. Transmission electron microscopy (TEM) image of the MIPN (B) along with the corresponding inverted image (C) (Ali, Mukhopadhyay, & Jana, 2019).

wide linear response ranging from 0.01 mg·L^{-1} to 10 mg·L^{-1}, low LOD of 1.80 μg·L^{-1} and good reproducibility. This research suggested a promising approach for food analysis and safety verification (Wu et al., 2019). Yang Liu et al. introduced a highly conductive sugarcane derived biochar nanoparticle into a tyrosinase nanosensor to act as a transducer and signal enhancer for the sensitive sensing of BPA. This nanosensor was successfully used in the on-site detection of BPA spiked ground water, and received a high recovery comparable to HPLC (Liu et al., 2019a, 2019b).

Tyrosinase is sensitive to its detection conditions including the temperature and pH. Therefore, Lingxia Wu et al. (Wu et al., 2020) prepared a tyrosinase nanocapsule to improve the stability of tyrosinase and therefor to establish a stable ultrasensitive BPA sensor. A low LOD of 12 nM and a wide linear range of 0.05–2 μM are obtained. In the detection of real samples, the BPA content in water bottles was detected to be 7.8 μg·g^{-1}, while no BPA was fund in coffee spoon. Those results together with the high recoveries in spiked samples detection indicated that this biosensor is a reliable tool for rapid detection of BPA in real food contact materials.

From the above studies, we can find that materials like MOFs, metal and carbon nanomaterials are usually utilized for the design of enzyme sensors to provide enzyme loading or enable it higher electroconductibility and catalytic activity. However, the enzyme-based BPs sensors often suffer from poor reproducibility, short shelf time and require well-trained personal to operate. Synthetic biomimetic enzyme sensors could overcome the shortcomings and have been widely used in the detection of many other molecules like $O_2^{\bullet-}$ and glucose. Therefore, searching and developing BPs biomimetic enzymes for in situ determination of BPs in food is of great significant.

2.3. Molecularly imprinted electrochemical sensors

In molecularly imprinted polymers (MIP), functional monomers along with target molecules and cross linkers are polymerized together, and target molecules are later eluted, leaving imprinted cavities on the polymer matrix (Dhanjai et al., 2018). The size and 3D structure-match enables selective recognition to target molecules (Zhang, Zhu, Wang, Xue, & Wang, 2017). In the last few years, MIP has caught more and more attentions of researchers because of its superior advantages, large surface area, high thermal stability and strong anti-interference ability (Ali, Mukhopadhyay, & Jana, 2019; Dhanjai et al., 2018), leading a wide usage of MIP in BPs electrochemical sensors (Table 1).

Although high selectivity, the catalytic ability of MIP is limited. Nanomaterials like graphene, carbon nanotubes (CNTs) and AuNPs are usually combined with MIP in order to improve the sensitivity and specificity of MIP-based electrochemical sensors (Rao et al., 2018). For example, Benzhi Liu et al. fabricated a MIP-based electrochemical sensor of BPA. Multiwalled carbon nanotubes (MWCNTs) were acrylamide-functionalized to work as a carrier to synthesize MIP (Liu et al., 2019). The acrylamide can induce the polymerization reaction on the surface of MWCNTs, increasing the number and density of the imprinted cavities. The sensor was successfully employed to determine BPA in plastic food contact materials.

Similarly, Haydar Ali et al. reported a molecularly imprinted nanocomposite (MIPN) that composed by polyacrylate, β-cyclodextrin and reduced graphene oxide (rGO) with a structure of 3D network toward the detection of BPA in water bottles and drinking water (Ali, Mukhopadhyay, & Jana, 2019) (Fig. 4). BPA can be selectively captured via a host–guest complexation by β-cyclodextrin and rGO. Under optimized conditions, the prepared electrode displayed a linear range from 0.02 μM to 10 μM and a LOD of 8 nM (Ali, Mukhopadhyay, & Jana, 2019). Yang's group fabricated a BPA electrochemical sensor using a molecularly imprinted chitosan-acetylene black composite film. It was successfully employed to detect BPA in unspiked and spiked drinking water samples (Tan et al., 2016).

2.4. Nanomaterials-based electrochemical sensors

The recognition elements aptamer, enzyme or MIP were mainly worked for selective and specific detection, while most of the above electrochemical sensors of BPs are tend to combined with various nanomaterials aim to acquire higher detection performance, because of the strong catalytic activity, large surface areas, fast electronic transfer or stability of nanomaterials (Yang et al., 2014). In recent years, the

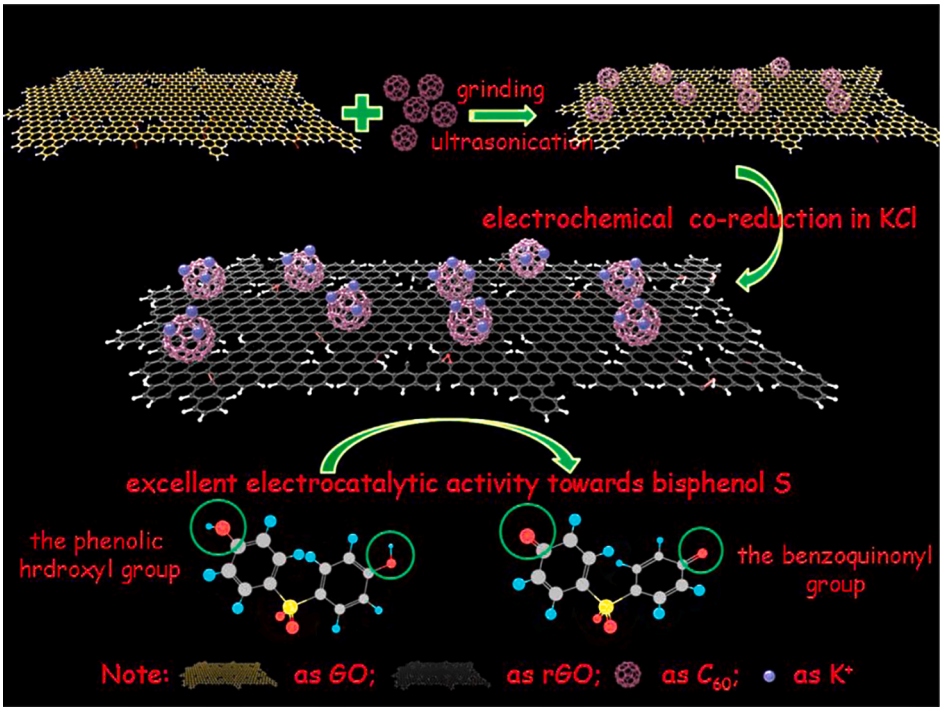


Fig. 5. Schematic diagram of the preparation process of the rGO-rC_{60} nanocomposite (Zhu et al., 2016).

aptamer, enzyme or MIP-free sensors with simple steps but high detection abilities are booming (Hashemi et al., 2019).

BPs are electroactive molecules, but their direct sensing is limited by high overpotential. Functional nanomaterials (graphene, CNTs and AuNPs et al.) can significantly enhance the electrocatalytic activity, lower the overpotential, increase the conductivity and response signals (Zheng et al., 2016). Carbon nanomaterials, metal nanomaterials and metal oxide nanomaterials which are well known catalysts with unique structures or components are summarized as follows.

2.4.1. Carbon nanomaterials based electrochemical sensors

Graphene as a superior carbon material with large contact surface area, high electric conductivity, single-atom thickness and wide electrochemical window has shown great promising applications in BPs detection. In 2016, we exploited an electrochemically co-reduced 3D GO-C_{60} (rGO-rC_{60}) based BPS sensing platform (Fig. 5), with which the rapid in site determination of BPS in milk samples was realized (Zhu et al., 2016).

CNTs are also extensively studied carbon materials. For example, carboxylated MWCNTs were succesfully applied to detect BPA in food package (Li et al., 2010). A carboxylic acid functionalized carbon black-MWCNTs (CB/f-MWCNTs) (Thamilselvan, Rajagopal, & Suryanarayanan, 2019) were adopted to enable the electrode with extraordinary properties like enhanced adsorption capacity, high electrocatalytic activity except enormous surface area and high conductivity. Peihong Deng et al. (Deng et al., 2013) presented a graphene modified acetylene black paste electrode to carry out sensitive direct determination of BPA. Thanks to the unique structure and extraordinary properties of graphene and acetylene black, the fabricated sensor displayed a low LOD of 0.6 nM and a wide range from 0.8 nM to 0.1 mM. Its practical analytical ability was proved by the successful usage in the measurement of BPA contained in unspiked food contact materials. The carbon nanomaterials-based BPs sensors are shown in Table 1.

2.4.2. Metal nanomaterials based electrochemical sensors

Metal nanomaterials, especially noble metal nanoparticles like platinum nanoparticles (PtNPs) (Zheng, Du, Wang, Feng, & Wang, 2013), palladium nanoparticles (PdNPs) (Su et al., 2017) and AuNPs (Yan et al., 2015) have been considered as ideal materials for the preparation of effective electrochemical sensors for their fascinating chemical and electronic properties (Mahmoudi et al., 2019b; Dhanjai et al., 2018). Recently, researchers have attempted to use cost-effective non-precious metals as alternative of noble metals to get more perfect electrochemical sensors.

Wang's group reported a nickel nanoparticles/nitrogen-doped carbon nanosheet (NiNPs/NCN) synthesized by one-step pyrolysis of nickel-1,3,5-benzenetricarboxylic acid MOFs (Wang et al., 2020). The nanocomposite was demonstrated to have large specific surface area, good electrical conductivity, strong electrocatalytic activity, and high anti-fouling ability, which lead to high sensitivity and improved reproducibility in BPA detection. Two linear pulse voltammetry (DPV) responses can be obtained in the concentration ranges of 0.1–2.5 μM and 2.5–15.0 μM, the electrochemical sensor was finally utilized to determine BPA in milk.

Besides, this group fabricated a copper-based Cu-MOFs that exhibited extraordinarily high surface areas, tunable structure, ultra-high porosity and naked active sites (Huang et al., 2020; Li et al., 2018). The Cu-MOFs based electrochemical sensor was utilized to determine BPA in plastic food contact materials. Xiaozhou Huang et al. designed a BPA electrochemical sensor based on bimetallic Ce-Ni-MOFs to measure the content of BPA in different brands of drinking water, and a satisfying recovery from 97.4 to 102.4% is acquired (Huang et al., 2020).

Mohammad Malakootian et al. prepared a $FeNi_3$/CuS/BiOCl nanocomposite that was used to modified on carbon paste electrode for BPA sensing (Malakootian, Hamzeh, & Mahmoudi-Moghaddam, 2020). The $FeNi_3$/CuS/BiOCl-based BPA sensor shows an excellent performance including high selectivity and sensitivity, good repeatability and stability, low LOD, which enable it to be used for the determination of BPA in unspiked food samples like tuna fish, tomato paste, apple and corn.

2.4.3. Metal oxide nanomaterials based electrochemical sensors

Apart from the above metal nanomaterials, metal compound like metal sulphide, metal carbides, metal nitrides, metal oxide and metal phosphides are developed. Among them, metal oxide with low toxicity and simple preparation are the most used metal compounds in sensors (Ghazizadeh, Afkhami, & Bagheri, 2018). Qin Liu et al. designed a electrochemical BPA sensors based on the glassy carbon electrode (GCE) decorated with $CoFe_2O_4$ nanoparticles which were synthesized by a sol–gel combustion method (Liu et al., 2020). A linear calibration curves range from 0.05 μM to 10 μM, and a LOD of 3.6 nM are obtained. The sensor was successfully utilized to determine BPA in milk, tap water and plastic food contact materials.

It is worth noting that the combination of different electrocatalytic nanomaterials will exhibit their respective advantages, improving the sensitivity and selectivity of electrochemical sensors, making the assay results more satisfactory. Kwanele Kunene et al. Modified MWCNTs with laccase enzyme immobilized by silver doped zinc oxide nanoparticles (Kunene, Sabela, Kanchi, & Bisetty, 2018). This nanocomposite was combined with screen printed electrodes for the detection of BPA. The unique composite of this nanocomposites helped to reduce the charge transfer resistance and thereby improved the sensitivity of BPA detection. This proposed biosensor was able to adequately quantify BPA in plastic bottles.

Qiong Wang et al. designed an ionic liquids@hollow porous spherical Ni-loaded $CdFe_2O_4$ (ILs@HPS-Ni/$CdFe_2O_4$) with high catalytic activity to realize the oxidation of BPS and BPAP (Wang et al., 2017). The ILs@HPS-Ni/$CdFe_2O_4$ sensor shows a good anti-fouling ability, fast electron transfer and large electrochemically active surface. The detection limits (S/N = 3) for BPS and BPAP are 5.37×10^{-9} M and 4.55×10^{-9} M, respectively.

Even most of the reported nanomaterial-based BPs electrochemical sensors have exhibited promising potential in real samples detection. The development of acceptable reference nanomaterial-based methods is still challenging, because of the two reasons: (I) Many interferent compounds that easier to oxidize than bisphenols might existed in real food samples. (II) Most of the nanomaterials have general electrocatalytic abilities for many molecules, which might decrease the selectivity of BPs determination. Aiming at these problems, preliminary treatments of samples or searching novel nanomaterials with high selectivity for BPs should be necessary, moreover, combining the recognize elements like aptamer, MIP and enzyme with nanomaterials would also be a useful way for the real sample detection. Hanbing Rao et al. proposed a MIP electrochemical sensor based on graphene quantum dots (GQDs) coated hollow nickel nanospheres. In the determination of BPS in mineral water and extraction solution of plastic samples, this sensor shows accurate results comparable to HPLC, providing a reliable method to monitor BPs in real food samples.

3. Conclusion and perspective

BPs pose enormous threats to human beings, it is necessary to develop methods for rapid and sensitive detection of BPs in food or food contact materials. Among all the developed BPs detection technologies, electrochemical sensors can be considered as the most promising method. Different types of BPs electrochemical sensors categorized according to recognition elements are discussed in this paper: aptamers, enzymes, MIP and nanomaterials based electrochemical sensors, of which the aptamer and enzyme are of great selectivity, but limited to relatively weak stability and repeatability since they are easily inactivated to temperature, pH or self-inhibition. The MIP inherits the inactivity characteristic of polymers, rending it high stability, selectivity and good repeatability and reproducibility. But the poor conductivity of MIP

restricts its use in sensitive determination of analytes. Nanomaterials have shown great potentials in the sensitive detection of BPs, expressing good repeatability and reproducibility, but their general catalytic ability to various molecules limited their selectivity. Therefore, composite materials of nanomaterials and aptamer, enzyme or MIP have emerged and are becoming a reliable method for the sensitive and selective detection of BPs in real food samples.

Even though the progress in developing BPs sensors is convincing, improvements in real samples analysis is still need to be focused. Designing and developing detection platform with better stability, sensitivity and selectivity are one useful way. Further, novel sensing system might simplify the operations steps and help to realize real time on-site detection and motivate the commercial viability of the electrochemical sensors. For example, developing portable and flexible sensing devices using functional materials, flexible electrodes or portable electrochemical system might make the real time on-site detection possible. Finally, for different food systems, applicable sensors should be picked to acquire accurate and precise testing. For baby bottles and sippy cups, in which the usage of BPA has been banned, electrochemical sensors with high sensitivity and low detection limit are needed, while for canned food or beverages, methods with detection limit lower than the banned limitation, 0.05 $mg{\cdot}kg^{-1}$ or 2.5 $mg{\cdot}kg^{-1}$, is enough.

This study presents a guideline for precise detection of BPs in food samples, the summarized fabrication methods and perspective of electrochemical sensors also provide a reference for the development and advancement in the relevant fields of research, like the monitoring of pesticides and antibiotics. However, the review is mainly focused on the electrochemical detection of bisphenols content in food, there is still extensive researches spaces in the detection of BPs existing in other systems. Additionally, except the electrode materials, the electrochemical sensing form and electrode types like microelectrodes, fiber electrodes are also should be summarized in the future work.

CRediT authorship contribution statement

Yuhuan Zhang: Project administration, Conceptualization, Writing - review & editing, Formal analysis. **Yanan Lei:** Writing - original draft, Software. **Hao Lu:** Software, Funding acquisition. **Lin Shi:** Writing - review & editing. **Peng Wang:** Funding acquisition, Writing - review & editing. **Zeshan Ali:** Funding acquisition, Formal analysis. **Jianke Li:** Conceptualization, Funding acquisition, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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