Chapter 9
Nanotechnology-Based Detection and Remediation of Mycotoxins for Food and Agriculture Applications

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Contents

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V. Kumar et al. (eds.), *Nanosensors for Environment, Food and Agriculture Vol. 1*, Environmental Chemistry for a Sustainable World 60,
https://doi.org/10.1007/978-3-030-63245-8_9

Abstract Mycotoxins are highly toxic secondary metabolites produced by fungi, which may contaminate a large variety of food and feed commodities. Among them, the aflatoxins, deoxynivalenol, zearalenone, fumonisins, and ochratoxins are the most common contaminants posing a serious threat to human and animal health. Given that traditional mycotoxin detection methods have been shown to be laborious and time consuming, interest in developing reliable and rapid mycotoxin detection methods has increased during recent decades. Herein, we review emerging nanotechnology-based methods, including gold nanoparticles, magnetic nanoparticles, and quantum dot-based sensors, which have been developed to reliably and efficiently detecting mycotoxins in food and feed commodities. We also summarize recent technologies used to remove mycotoxins via adsorption and photocatalytic degradation. As our review illustrates, the emerging use of nanotechnology offers a reliable and cost-effective means to prevent mycotoxin contamination in food and feed commodities, which could reduce health risks to consumers.

Keywords Mycotoxins · Food contamination · Nanotechnology · Nanosensors · Gold nanoparticles · Quantum dots · Magnetic nanoparticles · Photocatalytic degradation

9.1 Introduction

Mycotoxins are highly toxic secondary metabolites produced by fungi. They can invade a wide variety of agricultural crops while still on the field or afterwards during their processing in food and feed production chains. The presence of mycotoxins in agricultural commodities is high, with an estimated 25% of the world's crops being contaminated by molds or fungi, especially toxigenic species that belong to the genera Penicillium, Aspergillus, and Fusarium (Alshannaq and Yu 2017). At present, about 175 different mycotoxins have been identified in the food and feed commodities grown in outdoor and indoor environments (Bhat et al. 2010). Given their prevalence, mycotoxins have become a health hazard to both humans and animals worldwide (Fig. 9.1).

Based on possible dietary exposures together with their toxicity, aflatoxins (AFs), citrinin, patulin, ochratoxins A (OTA), zearalenone (ZEA), fumonisins, and trichothecenes are of a high concern due to their known health effects (Santos Pereira et al. 2019; Smith et al. 2016). Typical structures, source of contamination, food products contamination, health effects on mammalian system, and country-wise maximum tolerable levels of a wide range of mycotoxins are represented in Table 9.1. Risk assessments of mycotoxins in food are governed by the Joint Expert Committee of the Food and Agriculture Organization (JECFA) of the United Nations (FAO) and World Health Organization (WHO). The international standards are established by the Codex Alimentarius Commission, which lists maximum levels for various mycotoxins in foods (Jukes 2000; Organization 1999).

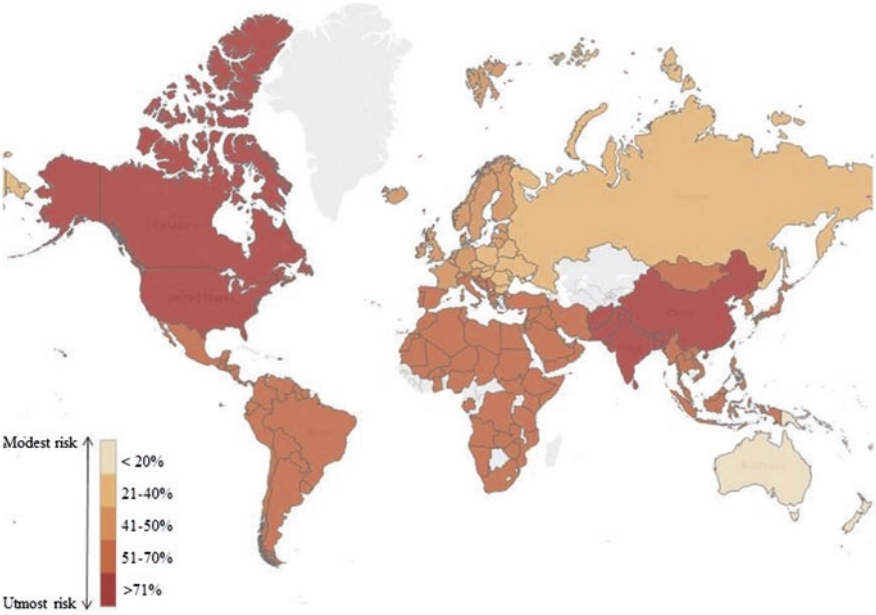
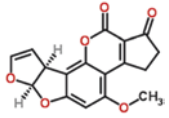
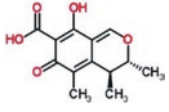
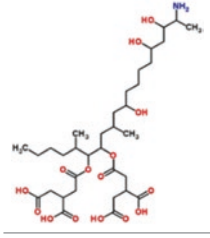


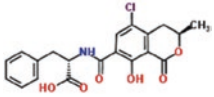
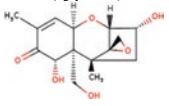
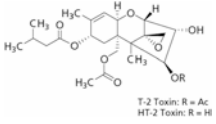
Fig. 9.1 Estimated worldwide health risk caused by mycotoxins during 2018. Data source: World Nutrition Forum/Database (BIOMIN, 2019)

The traditional methods of mycotoxin analysis involve more complex sample preparation and longer detection times combined with the usage of highly sophisticated analytical instruments (Vidal et al. 2013; Zhang et al. 2018a). Nanotechnology has gone through a booming development since 1981. The usage of nanoparticles in sensing applications are accurate with high precision resulting in signal amplification due to their unique characteristics, such as high surface to volume ratio (Tothill 2011). Further the integration of nanomaterials not only enhances the sensitivity in detection of toxins, but they are highly selective, cost effective, and portable and a rapid response can be obtained (Malhotra et al. 2014). A wide-range of nanomaterials comprising gold nanoparticles, silver nanoparticles, quantum dots (QDs), magnetic nanoparticles, graphene oxide, carbon nanohorns, carbon nanotubes (CNTs), and TiO_2 nanoparticles, have been used for the detection of different kinds of mycotoxins.

Till date many reviews have been published in sensing and detection of mycotoxins using nanotechnology (Goud et al. 2018; Horky et al. 2018; Ingle et al. 2020; Zeng et al. 2016). This is a comprehensive review including the various sensing applications with some of extensively used nanoparticles, such as gold, QDs, and magnetic nanoparticles. Here, we review advances from the last 5 years in the use of nanomaterials for the detection of mycotoxins in food and agriculture. This review also summarizes the recent technologies employed for the removal and the photocatalytic degradation and removal of mycotoxins. A literature search for the years

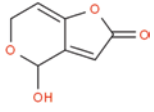
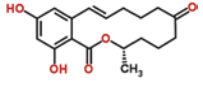
Table 9.1 Mycotoxin sources, occurrence in foods, and health effects

Mycotoxin type/ Typical structure	Source	Food products contaminated	Health effects on mammalian system	Country-wise maximum tolerable levels		Refs.
				Country	Tolerable levels (μg kg^{-1})	
Aflatoxins (B1, B2, M1, M2, G1, and G2) 	*Aspergillus flavus*, *Aspergillus nominus*, *Aspergillus parasiticus*	Maize, oil seeds, nuts, dried fruits, cereals, spices, milk, and milk products including infant food	Carcinogenic, acute hepatitis, impaired immune system	Australia, Canada, Europe	14–15	chuan Li et al. (2011), Wang et al. (2011) and Ye et al. (2010)
				Nigeria, New Zealand		
				South Africa		
				USA, Brazil	20	
				India	30	
Citrinin 	*Aspergillus carneus, Aspergillus niveus, Aspergillus terreus, Penicillium citrinum, Penicillium verrucosum, Penicillium expansum*	Oats, rice, corn, beans, fruits, fruit and vegetable juices, herbs, and spices	Nephrotoxicity, hepatotoxicity, embryotoxicity, cytotoxicity, immunotoxicity, carcinogenicity			(Arévalo et al. (2011) and Flajs and Peraica (2009)
Fumonisins (B1, and B2) 	*Aspergillus alternaria, Fusarium anthophilum, Fusarium dlamini, Fusarium moniliforme, Fusarium naphiformel, Fusarium nygama, Fusarium proliferatum, Fusarium verticillioides*	Maize, rice, sorghum, cereals, green gram	Carcinogenic, nephrotoxic, hepatotoxic, teratogenic	Europe, Turkey, Norway, Switzerland	800–4000	Abdul Kadir and Tothill (2010) and Ghali et al. (2009)
				USA	2000–4000	
				Brazil	2000–5000	

OTA 	*Aspergillus alliaceus, Aspergillus auricomus, Aspergillus carbonarius, Aspergillus glaucus, Aspergillus melleus, Aspergillus niger, Aspergillus ochraceus, Penicillium cyclopium, Penicillium verrucosum, Penicillium viridicatum*	Maize, rice, rye, cereals, dry fruits, coffee, wine, beer, grape, juice, spices, licorice	Mild liver damage, nephrotoxicity, and immune suppression	Brazil	20–30	Heurich et al. (2011), Pfohl-Leszkowicz et al. (2015), Barthelmebs et al. (2011) and Wu et al. (2011)
				China, Kenya	5	
				Europe, Egypt	2–10	
				India	20	
				Nigeria, Russia	5	
				Uruguay	50	
Trichothecenes DON, 3- or 15-Ac-DON, NIV (type B) 	*Fusarium graminearum, Fusarium sporotrichioides, Fusarium poae, Fusarium equiseti*	Cereals, bakery products	Weight loss, diarrhea, vomiting, gastrointestinal hemorrhaging, immune-depressants, dermal necrosis mutagenic, neurotoxic	Europe	500–1750a	Chehri and Godini (2017), Li et al. (2018b) and Maragos (2012)
				Brazil	750–3000a	
				Russia	700–1000	
				Canada, China, India, Japan, USA	1000	
Trichothecenes T-2, HT-2 (type A) 	*F. sporotrichioides*	Corn, wheat, barley, oats, rice, rye	Weight loss, diarrhea, vomiting, gastrointestinal hemorrhaging, immune-depressants, dermal necrosis mutagenic, neurotoxic	Europe	Not permitted	Authority et al. (2017) and Pleadin et al. (2018)
				Russia	50–100a	

(continued)

Table 9.1 (continued)

Mycotoxin type/ Typical structure	Source	Food products contaminated	Health effects on mammalian system	Country-wise maximum tolerable levels		Refs.
				Country	Tolerable levels (μg kg^{-1})	
Patulin 	*Aspergillus clavatus, Aspergillus longivesica, Aspergillus terreus, Penicillium expansum, Penicillium griseofulvum, Byssochlamys sp.*	Apple juice and solid apple products	Genotoxicity, teratogenicity, cancer	Brazil, China, Europe, India, Japan, Kenya, Nigeria, Russia, South Africa, USA	50	Erdoğan et al. (2018) and Zhong et al. (2018)
Zearalenone 	*Fusarium culmorum, Fusarium crookwellense, F. equiseti, F. graminearum, F. sporotrichioides*	Corn, wheat, wheat flour, bread, cereals, noodles, rice, barley, oats, sorghum, walnuts, milk, corn beer, meat, animal feed products	Estrogenic activity (infertility, vulval edema, vaginal prolapse, mammary hypertrophy in females, feminization of males	Europe	75–400a	Panini et al. (2010)
				Brazil	200–1000a	
				China, Russia, Chile	200,000	

from 2014 to 2019 was conducted in the PubMed (https://www.ncbi.nlm.nih.gov) and Scopus (https://www.scopus.com) databases using the keywords "nanoparticles, mycotoxin detection." Additionally, three categories of nanomaterials were searched specifically: "gold nanoparticles," "magnetic nanoparticles," and "quantum dots."

9.2 Nano-sensors for the Detection of Mycotoxins

At present, there is an increasing attention on nanotechnology since it has generated novel advantages across the range of areas comprising the food industry. Accordingly, nanotechnology has revealed new tasks for novelty in food production at a quick rate. Quality and safety of foods are a key component of public health, and with growing public awareness, customers are now demanding foodstuffs that are devoid of any contaminants (Hamad et al. 2018; Pal 2017; Pathakoti et al. 2019; Pradhan et al. 2015).

Due to their staggeringly small size, nanomaterials show specific physical and chemical features. Nanosensors are described as any chemical, biological, or surgical sensory point utilized to expose the nanoparticles to the microscopic world (Yu et al. 2018). Nanosensors utilize several nanomaterials that can identify toxins in sustenance at precise low levels, in the midst of handling or processing of foods (Sonawane et al. 2014; Willner and Vikesland 2018). Thus, in nanosensors, nanomaterials are employed in an analytical device, which is an improved version of a chemical sensor or biosensor. A biosensor works on two basic principles, biological recognition, and sensing. In general, nanosensors or nanobiosensors enhance at the activity by the integration of nanostructures in the sensing component for enhanced output. The basic structure of nanosensor and working principal is schematically shown in Fig. 9.2. These nanostructures enhance the activity both at bioreceptor level and the transducer level. Moreover, the transducer aids in fabrications of sensor with the use of various nanoparticles, such as metal oxide nanoparticles, quantum dots, magnetic nanoparticles, and carbon nanoparticles. Due to their staggeringly small size, nanomaterials show specific physical and chemical features, such as superior optical, electrical, thermal properties, and high surface area, which improves the transducing capability to a large extent. Based on signal production, that is, based on transducer type, nanosensors can be three major types: (1) optical: as optical (colorimetry, fluorescence, luminescence, surface-enhanced Raman scattering (SERS), surface plasmon resonance (SPR) and others), (2) electrochemical (amperometry, conductimetry, potentiometry, and voltammetry), and (3) Piezoelectric (quartz crystal microbalance).

9.2.1 Gold Nanoparticles for the Detection of Mycotoxins

A variety of biochemical and electrochemical assays based on Au nanoparticles have been described for the detection of mycotoxins. The most important and advantageous properties of gold nanoparticles for their usage in designing the

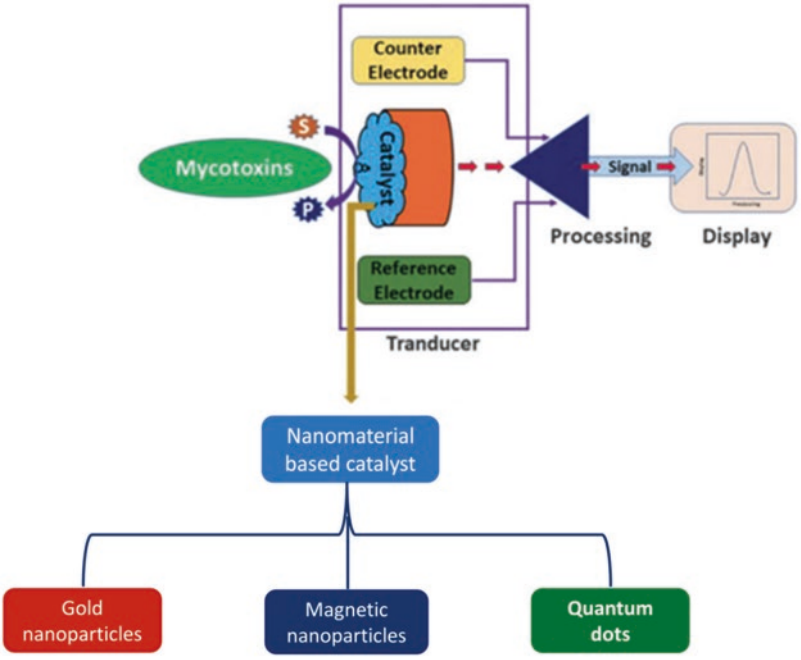


Fig. 9.2 Schematic representation of nanosensor and its working. modified from Goud et al. (2018), Copyright (2018) Elsevier

sensors to detect toxins include localized surface plasmon resonance (SPR) bands and enhanced scattering properties (Lara and Perez-Potti 2018). SPR imaging (SPRi) is an evolving label free semi-quantitative method for the sensitive detection of mycotoxins, which is highly reliable (Joshi 2017). Hossain and Maragos Hossain and Maragos (2018) described a gold nanoparticle based SPRi for the detection of Fusarium toxins such as DON, ZEA, and T-2 in wheat samples. This biosensor was amplified more than 12–90-fold with use of secondary antibody Ab2 with Au nanoparticles. Further, these antigen coated biochips are highly durable with short analysis time, are cost effective, and can be used for at least 46 cycles. In another study, Kong et al. (2016) have developed an Au nanoparticle-based immunochromatographic strip system for the detection of 20 different types of mycotoxins including ZEAs, DONs, T-2 toxins, AFs, and fumonisins (Fig. 9.3).

Similarly, AFs including B1, B2, M1, G1, and G2 in sustenance were identified depending upon a lateral flow strip (Santos et al. 2017). A monoclonal antibody (mB6 mAb), which has high specificity to AFs, was created to develop an immunochromatographic strip test, where the gold nanoparticles were coated with this antibody for detection. This strip consists of a control and test line, where the control line is coated with goat antimouse IgG and the test line containing AFB1 bovine serum albumin. In the strip, red gold nanoparticles form 1–2 pigmented lines on the

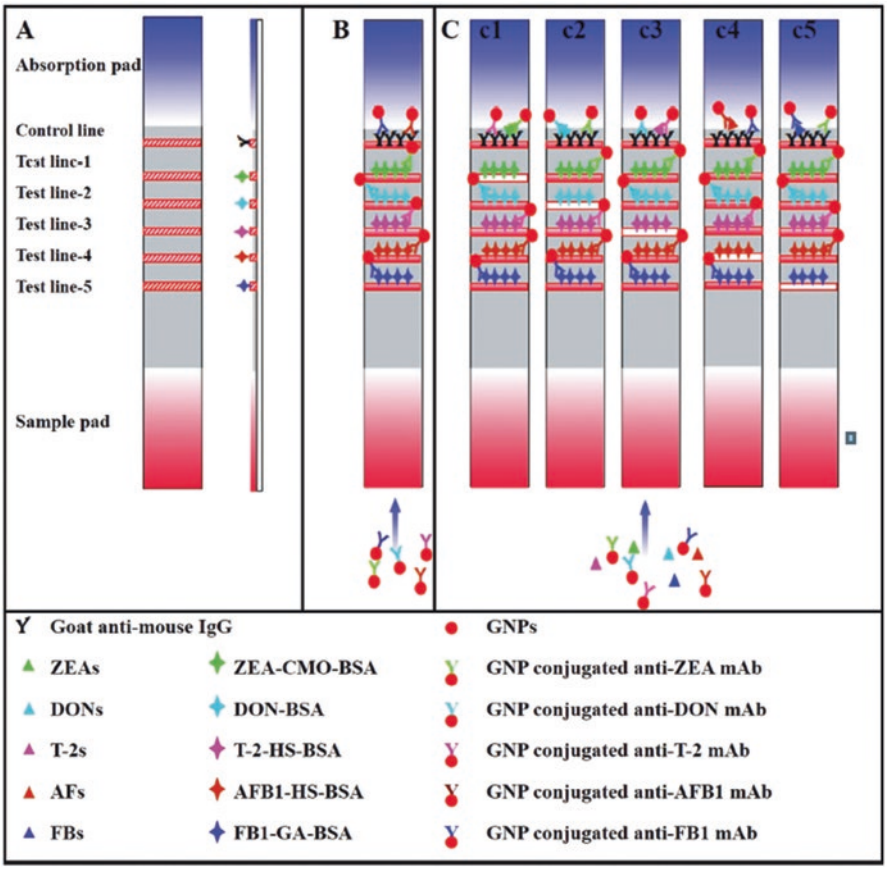


Fig. 9.3 (A) Immunochromatographic strip structure (B) Negative sample (C) Positive sample; c1: ZEAs; c2: DONs; c3: T-2s Toxin; c4: Aflatoxins; c5: Fumonisin B. Copyright (2016) Elsevier, (Kong et al. 2016)

membrane and the nanoparticles coated with anti-AFB1 as the signaling reagent for basic identification of AFB1.

A surface-enhanced Raman scattering (SERS) aptamer-based sensor was developed by using the gold@gold-silver nanostructures for the selective detection of OTA (Fig. 9.4). Magnetic nanoparticles (Fe_3O_4) were used for separation of toxin from solution, which is reusable. After magnetic separation, Raman signals were collected and this SERS probe can be used for the real time detection of OTA (Shao et al. 2018). Table 9.2 represents a wide range of methodology for sensor development using gold nanomaterials for the detection of different types of mycotoxins.

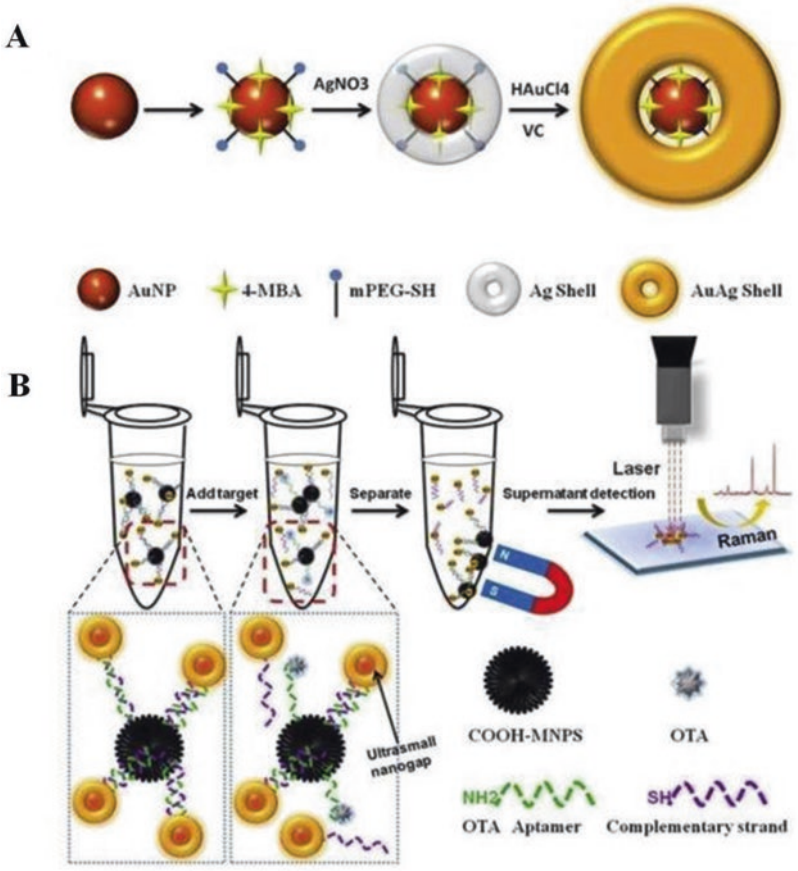


Fig. 9.4 Synthesis of Au@AuAg NNSs (A). Schematic illustration of SERS aptsensor-based on Au@AuAg NNSs-magnetic nanoparticles for OTA detection. Copyright (2018) Elsevier (Shao et al. 2018)

9.2.2 Magnetic Nanoparticles for the Detection of Mycotoxins

Superparamagnetism is one of the most important properties of magnetic nanoparticles. The superparamagnetic nanoparticles can be easily dispersed in aqueous solution, remain stable when coated with an appropriate layer, and are able to form ferrofluid. Wide ranges of applications of superparamagnetic nanoparticles are reported. Various methods of detection of mycotoxins by employing magnetic nanoparticles as sensors are represented in Table 9.3. A study by Gan et al. (2013) described the electrochemiluminescent immunoassay for the detection of mycotoxin, aflatoxin-M1 in milk samples. In their study, magnetic nanocomposites were prepared initially by immobilization of Fe_3O_4 nanoparticles onto graphene oxide, which is used as an adsorbent. Later on, aflatoxin-M1 antibody was attached to

Table 9.2 Detection of mycotoxins by using gold nanoparticles as sensors

Mycotoxin	Electrode/sensor	LOD or recovery (%)	Sample or matrix	Refs.
Patulin	Molecular imprinted polymer-SERS technique	96–108%	Fruit samples	Wu et al. (2019)
Ochratoxin A	AuNP@Cu-Co Purssian blue analogue-based electrochemical conductivity assay	5.2 fg mL^{-1}	Spiked watermelon juice	Gu et al. (2019)
Patulin	AuNP-Black phosphorus nanosheets-electrochemical aptasensing	0.03 nM	Apple juice samples	Xu et al. (2019)
Fuminosin B1 and DON	Indium Tin oxide@ Polydimethylsiloxane electrodes functionalized with Au nanoparticles and anti-FB1 and anti-DON antibodies	0.3–140 ppb 0.2–60 ppb	Ground corn extract	Lu and Gunasekaran (2019)
ZEA	Calorimetric assay: use of an aptamer and Au nanoparticles with peroxidase like activity	10 ng mL	Corn and corn oil	Sun et al. (2018)
AFB1	Optical waveguide light mode spectroscopy (OWLS): signal enhancement by increasing the sensor surface through immobilization of Au nanoparticles on OWLS immunosensor chip	0.01–10 ng mL^{-1}	Hungarian paprika samples	Adányi et al. (2018)
Fuminosin B1 and B2	Fluorescence quenching immunoassay based on Au nanoparticles and a recombinant epitope-mimicking fusion protein	1.1 ng mL^{-1}	Wheat samples	Peltomaa et al. (2018)
Fuminosin B1, B2, and B3	Indirect competitive enzyme-linked immunosorbent assay: Au nanoparticles–mercaptoundecanoic acid@ horseradish peroxidase—goat anti-mouse IgA	0.078μg mL^{-1}	Maize	Li et al. (2018c)
AFB_1	L-arginine- 6-aza-2-thiothymine—Au nanocluster-based photoluminescence enzyme immunoassay	3.2 pg mL^{-1}	Peanut	Wang et al. (2018)
OTA	Detection based on signal amplification by Exonuclease III (Exo III) and fluorescence quenching by Au nanoparticles	4.82 nM	Spiked red wine	Zhao et al. (2018)
Fuminosins	Lateral flow immunoassay: CdSe/ZnS QD-BSA-FMB1@AuNP-Ab	1 ng mL^{-1}	Maize flour samples	Anfossi et al. (2018)
DON; ZEA; T2/HT2-toxin	Multiplex lateral flow immunoassay: monoclonal antibodies (mAb) conjugated with CdSeS/ZnS QDs or colloidal Au nanoparticles	80–1000μg kg^{-1}	Wheat samples	Foubert et al. (2017)

(continued)

Table 9.2 (continued)

Mycotoxin	Electrode/sensor	LOD or recovery (%)	Sample or matrix	Refs.
AFB1	Label-free electrochemical	8 pg mL^{-1}	Corn flakes	Chauhan et al. (2016)
	Quartz crystal microbalance			
	Based immunoassay: α-AFB1/ cysteamine/Au nanoparticles/ hexanedithiol (HDT)/Au immunosensor			
ZEA	Immunochromatographic assay: antibody immobilization on ablative Au nanoparticles	0.1 ng mL^{-1}	Standard ZEA	Urusov et al. (2015)
OTA	Label-free aptamer-based assay: cationic polymer poly diallyldimethylammonium chloride@Au nanoparticles	0.009 ng mL^{-1}	Liquor samples with OTA	Luan et al. (2015a)
OTA	SERS labels embedded Ag@Au core–shell nanoparticles	0.006 ng mL^{-1}	Spiked mycotoxins on negative maize meal	Zhao et al. (2015)
AFB1		0.03 ng mL^{-1}		
DON	Electrochemiluminescence electrodes based on nanoporous Cobalt/Co_3O_4 − Au/RuSi@ $Ru(bpy)_3^{2+}$	1 pg mL^{-1}	Wheat flour	Lv et al. (2015)
AFB1	AFB1-BSA conjugate modified magnetic beads were employed as capture probe and *anti*-AFB1 antibody-coated gold colloids were used as detection probe for the immunological recognition	7 pg mL^{-1}	AFB1 standard solutions	Wang et al. (2016b)
AFB1	SERS-based immunoassay using silica-encapsulated hollow Au nanoparticles and magnetic beads	0.1 ng mL^{-1}	AFB1 standard solutions	Ko et al. (2015)
DON	The electrochemical impedance spectroscopy of tris(bipyridine) ruthenium (II) chloride was used as a marker enhanced with Au nanoparticles-dotted 4-nitrophenylazo functionalized graphene nanocatalyst mediated in Nafion on a glassy carbon electrode	0.3μg mL^{-1}	Certified corn, wheat, and roasted coffee	Sunday et al. (2015)
AFB1	Label-free aptasensor using un-modified Au nanoparticles	0.025 ng mL^{-1}	AFB1 standard solutions	Luan et al. (2015b)
AFB2	NaCl-induced aggregation of aptamer-modified Au nanoparticles	25 pg mL^{-1}	AFB2 standard solutions	Luan et al. (2015a)

(continued)

Table 9.2 (continued)

Mycotoxin	Electrode/sensor	LOD or recovery (%)	Sample or matrix	Refs.
OTA	Immunochromatographic assay: OTA monoclonal antibody conjugated Au nanoparticles	0.25 ng mL^{-1}	Maize, wheat, soybean, and rice	Majdinasab et al. (2015)
AFB1	SPRi: antibody-conjugated Au nanoparticles on poly[oligo(ethylene glycol) methacrylate-co-glycidyl methacrylate]@modified SPRi chip	8 pg mL^{-1}	Spiked peanut samples	Hu et al. (2014)
OTA		30 pg mL^{-1}		
ZEA		15 pg mL^{-1}		
Fumonisin B1	Electrochemiluminescence aptosensor: Au NP–Iridium complex as nanoprobe	0.27 ng mL^{-1}	Spiked wheat flour	Zhao et al. (2014)

*LOD: Limit of detection

cadmium telluride quantum dots, which can be used as a signal tag. Finally, magnetic nanocomposites were used for the separation of aflatoxin-M1 from milk samples and detected using an immunoassay using an electrochemiluminescent signal. Further, this immunosensor is stable and can be regenerated after storing for two weeks at 4°C and its accuracy was about 95%.

9.2.3 Quantum Dots for the Detection of Mycotoxins

Physicochemical properties including enhanced fluorescence, narrow emission peaks, and high photostability of QDs are advantageous in developing the sensors for highly sensitive detection of mycotoxins. Some of the recent applications of quantum dots for mycotoxin detection are presented in Table 9.4. Fang et al. (2014) developed a novel molecularly imprinted optosensing material for selective detection of mycotoxin, ZON in cereal samples. The molecular imprinted polymer is based on ionic liquid stabilized cadmium selenium/ZnS QDs and the mechanism is based on fluorescence quenching.

A highly sensitive aptasensor for the detection of ochratoxin was developed with a combination of nanocomposites consisting of gold nanoparticles functionalized with silica coated Fe_3O_4 magnetic nanoparticles and CdTe QDs modified with graphene/gold nanoparticles. This novel aptasensor is ultrasensitive with detection limit at sub-picomolar level (Hao et al. 2016).

Zhang et al. (2017a) developed an optical sensor based on molecular imprinted polymer capped Mn-doped ZnS QD for detection of the mycotoxin, patulin, from apple juice. This nanosensor is based on a phosphorescence method, for determination of patulin from aqueous solutions, including the intended matrix, apple juice. Recently, Duan et al. (2019) developed multicolor CdSe/ZnS QD nanobeads with yellow, orange, and red luminescence for concurrent recognition of numerous

Table 9.3 Detection of mycotoxins by using magnetic nanoparticles as sensors

Mycotoxin	Electrode/sensor	LOD or recovery (%)	Sample or matrix	Refs.
OTA	SERS: Au@AuAg-magnetic nanoparticle	0.004 ng mL^{-1}	Red-wine samples	Shao et al. (2018)
ZEA	Aptamer-functionalized MNP @ fluorescence NaYF4: Ce/Tb nanoparticles	0.21 pg mL^{-1}	Maize and wheat	Niazi et al. (2018)
AFB1	Magnetic nanoparticle-filled amino-modified multi-walled carbon nanotubes (Fe_3O_4-MWCNTs-NH_2)	0.15 and 0.24 ng g^{-1}	Wheat flour samples	Li et al. (2018a)
ZEA				
Fumonisin B1	Magnetic reduced graphene oxide/nickel/platinum nanoparticles	0.70 ng mL^{-1}	Beer and wine	Molinero-Fernández et al. (2018)
ZEA; α-Zearalanol	Core–shell polydopamine coated magnetic nanoparticle	0.55–11.8μg L^{-1}	Cow, goat, sheep, and human milk	Socas-Rodríguez et al. (2018)
α-Zearalenol;				
β-Zearalanol				
β-Zearalenol;				
β-Zearalanol-10,10,11,12,12-d5				
AFB1	SERS based aptasensor: the amino-terminal aptamer conjugated magnetic-bead and the gold nanotriangles-DTNB@ Ag-DTNB nanotriangles	0.54 pg mL^{-1}	Peanut oil samples	Yang et al. (2017)
AFB1	Magnetic microspheres encoded with fluorescent nanocrystals	9 pg mL^{-1}	Spiked corn samples	Zhang et al. (2017b)
ZEA,	Core–shell poly(dopamine) magnetic nanoparticles	0.2–4.8μg L^{-1}	Milk Yogurt	González-Sálamo et al. (2017)
α-zearalanol,				
β-zearalanol,				
α-zearalenol,				
β-zearalenol zearalanone				
OTA	Fluorescence resonance energy transfer: quantum dot-labeled antibody to rhodamine-coated magnetic silica nanoparticles	0.8 pg mL^{-1}	Spiked human serum	Mahdi et al. (2016)
ZEA	Magnetic nanoparticles and chemiluminescent detection	0.04 ng mL^{-1}	Wheat	Hendrickson et al. (2016)
OTA	Chemiluminescence immunoassay: magnetic nanoparticles with targeted inhibition	1.39 pg mL^{-1}	Rice	Kim and Lim (2015)

(continued)

Table 9.3 (continued)

Mycotoxin	Electrode/sensor	LOD or recovery (%)	Sample or matrix	Refs.
AFB1	FRET-based fluorescence immunoassay: magnetic/silica core–shell as a signal intensifier	2×10^{-12} M	Spiked human serum	Kalarestaghi et al. (2015)
AFB1	Immunoenzyme assay: antibody immobilization on the surface of magnetic nanoparticles	20 pg mL^{-1}	Barley and maize extracts	Urusov et al. (2014)

mycotoxins such as ZEA, OTA, and fumonisin B1 from a corn matrix (Fig. 9.5). This is a qualitative method based on an immunochromatographic assay for visual detection of mycotoxins, which can be inferred with the naked eye under UV light with color differentiation.

9.2.4 Miscellaneous Nanomaterials Employed in Developing Sensors for the Detection of Mycotoxins

Zinc oxide nanoparticles are recognized for applications in immunosensing, due to their distinct characteristics, such as a high isoelectric point and higher binding energy and moreover being cost-effective and biocompatible (Ansari et al. 2010). For OTA detection, ZnO nanofilm was immobilized with rabbit immunoglobulin antibodies placed onto glass plate made of Indium tin oxide and bovine serum albumin for blocking nonspecific binding of OTA. This nanozinc immunoelectrode has amplified electrochemical signal and has an application for OTA and other mycotoxins (Ansari et al. 2010).

Similar to ZnO nanoparticles, nanostructured CeO_2 is biocompatible due to its non-toxicity and has attracted interest in development of biosensors. In addition, nano-CeO_2 has high chemical stability and mechanical strength. Kaushik et al. (2009a) described the fabrication of chitosan-nano-CeO_2 nanocomposites film deposited on indium tin oxide-coated glass substrate for the immobilization of r-IgGs and BSA. This immunoelectrode has a large surface, including greater electron transport and potential application for OTA detection and other mycotoxins such as AFB1 and citrining.

Likewise, a study by Kaushik et al. (2009b) reported the detection of OTA by integrating nanosilicon-chitosan nanocomposites film for r-IgGs loading, and this immunoelectrode has enhanced sensing properties. This could be due to the presence of chitosan-nano-SiO_2 composites due to greater surface area and higher electrochemical behavior. Similarly, a chitosan-TiO_2-nanoparticle-based immunosensor was developed for the recognition of OTA from *Aspergillus ochraceus* (Khan and Dhayal 2008). Electrochemical impedance spectroscopy (EIS) was used to determine the electroactivity of the bioactive electrode. This matrix has several advantages in biosensor application. Chitosan is cost-effective and has higher mechanical

Table 9.4 Detection of mycotoxins by using quantum dots

Mycotoxin	Electrode/sensor	LOD	Sample or matrix	Refs.
ZEA	Electrochemiluminescence aptasensor: Nitrogen doped graphene QDs on amine functionalized Ru(bpy)3 2+-doped silica nanoparticles surface	1 fg mL^{-1}	Corn flour	Luo et al. (2019)
Fumonisin B1	Fluorescence Enzyme linked immunoassay: mercaptopropionic acid–modified CdTe QDs	0.33 ng mL^{-1}	Corn	Lu et al. (2018)
DON	Fluorescent-labeled immunosorbent assay: monoclonal antibody+amino functionalized multishell QDs	12.2μg kg^{-1}	Maize	Zhang et al. (2018b)
AFB1	Glycine-enhanced photoluminescence assay: 3-mercaptopropionic acid and thiol-terminated methoxy polyethylene Glycol functionalized QDs	0.17–0.35 ng mL^{-1}	Chinese medicinal herbs	Zhang et al. (2018a)
OTA	Fluorescence resonance energy transfer: cerium oxide nanoparticles and graphene QDs aptosensor	2.5 pg mL^{-1}	Peanuts	Tian et al. (2018)
OTA	Direct competitive fluorescence linked immunosorbent assay: QD beads	0.028 pg mL^{-1}	Corn	Xiong et al. (2017)
			Coffee	
			Red wine	
OTA,	Electrochemical detection: CdTe or Pbs QDs coated on to silica sphere	10 pg mL^{-1}–10 ng mL^{-1}	Maize	Wang et al. (2017)
Fumonisin B1		50 pg mL^{-1}–50 ng mL^{-1}		
AFM1	Frit based immunoassay: red and green fluorescent CdSe/ZnS core/shell QDs immobilized in liposomes	0.02μg kg^{-1}	Spiked and natural milk samples	Jiang et al. (2017)
Pirlimycin		0.5μg kg^{-1}		
ZEA,	Multiplex immunochemical assay: InP/ZnS QDs encapsulated in silica shells	50 and 500μg kg^{-1}	Maize	Beloglazova et al. (2017)
DON			Wheat	
DON, ZEA, & AFB1	Immunoassay: colloidal QDs enrobed into silica shell	6.1 and 5.3	Cereals	Beloglazova et al. (2016)
		5.4 and 4.1		
		2.6 and 1.9		
OTA	Fluorescence enzyme linked immunosorbent assay: glucose oxidase-mediated fluorescence quenching of mercaptopropionic acid-capped CdTe QDs	2.2 pg mL^{-1}	Corn	Liang et al. (2016)
ZEA	Fluorescence enzyme linked immunosorbent assay: hydrogen peroxide-sensitive QDs	4.1 pg mL^{-1}	Corn	Zhan et al. (2016)

(continued)

Table 9.4 (continued)

Mycotoxin	Electrode/sensor	LOD	Sample or matrix	Refs.
AFB1	Photoelectrochemical immunoassay: CdTe QDs-modified photosensitive electrode	3.0 pg mL^{-1}	Spiked peanut samples	Lin et al. (2016)
DON	Immunoassay: silica-coated CdSe/CdS/ZnS core–shell QDs	20 ng mL^{-1}	Food and feed	Goftman et al. (2016)
OTA	Electrochemiluminescence assay: RuSi nanoparticles/CdTe QDs	3.0 fg mL^{-1}	Corn and human serum	Wang et al. (2016a)
OTA	Fluorescence immunoassay: hydrogen peroxide-sensitive CdTe QDs	0.05 pg mL^{-1}	Corn	Huang et al. (2016)
			Wheat	
			Rice	
ZEA	Immunochromatography assay: CdSe/ZnS QDs submicro beads	3.6 mg kg^{-1}	Corn	Duan et al. (2015)
DON, ZEA, AFB1, T2-toxin, Fumonisin B1	Fluorescent immunosorbent assay: QD nanolabels	3.2μg kg^{-1}	Cereals	Beloglazova et al. (2014)
		0.6μg kg^{-1}		
		0.2μg kg^{-1}		
		10μg kg^{-1}		
		0.4μg kg^{-1}		
AFB1	Fluorescent quenching assay: graphene oxide-aptamer-CdTe QDs	1.4 nM	Peanut oil	Lu et al. (2015)

*LOD: Limit of detection

strength and permeability. TiO_2 provides the biocompatibility and thereby provides longer life and improved stability to the electrode. This technique of conjugating chitosan with nanoparticles also provided the fastest response (25 s) with the lowest detection limit.

Recently, Goftman et al. (2016) synthesized the silica coated CdSe quantum dots to detect DON by the microemulsion method. Further, to increase the bioapplicability of QD@SIO_2 nanoparticles, they were modified using three diverse functional groups including amino, carboxyl, and epoxy groups and polyethylene glycol fragments. This developed fluorescence-labeled immunosorbent assay was used for rapid detection of mycotoxins, especially DON.

Carbon nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure (Saifuddin et al. 2013). Among them, single-walled CNTs (SWCNTs) due to their exceptional characteristics such as greater surface area, high electrical conductivity, and mechanical strength provide a compatible environment to preserve the enzyme activity, thereby enhancing the electrochemical signal amplification (Guo 2013; Singh et al. 2009). Zhang et al. (2016) fabricated an electrochemical immunosensor for detection of AFB1 from corn flour using the SWCNTs and chitosan composites. As SWCNTs are not water soluble, addition of chitosan enhanced the dispersion of SWCNTs. This immunosensor was centered on an indirect competitive assay using the primary antibody anti-AFB_1 and AFB_1-BSA immobilized on glass carbon electrode.

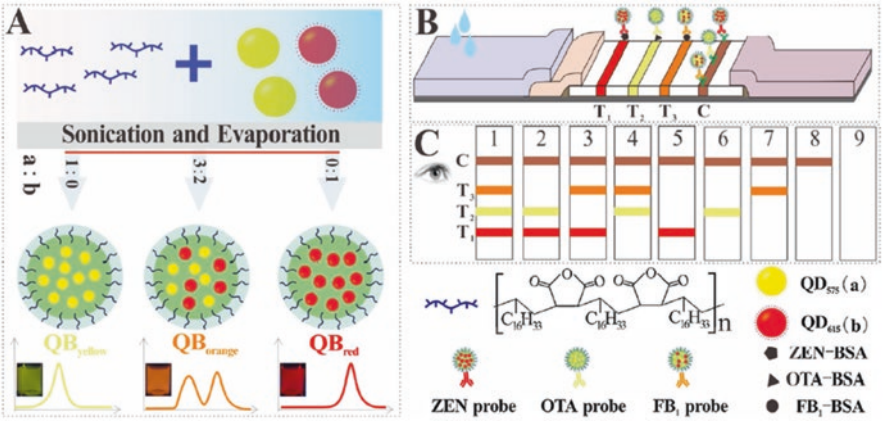


Fig. 9.5 Scheme 1. (**a**) Schematic representation for the synthesis of tricolor QB via using the emulsification evaporation method. (**b**) Schematic illustration of developed tricolor QB-based mICA, in which three T lines of T1, T2, and T3 were designed for the simultaneous detection of ZEN, OTA, and FB1, while one C line was set to indicate the validity of developed mICA, respectively. (C) Schematic presentation for the interpretation of test results. 1, negative; 2, ZEN (−), OTA (−), FB1 (+); 3, ZEN (−), OTA (+), FB1 (−); 4, ZEN (+), OTA (−), FB1 (−); 5, ZEN (−), OTA (+), FB1 (+); 6, ZEN (+), OTA (−), FB1 (+); 7, ZEN (+), OTA (+), FB1 (−); 8, ZEN (+), OTA (+), FB1 (+); 9, invalid. ("+" and "−" indicate the "positive" and "negative," respectively). Copyright from (2019) Elsevier (Duan et al. 2019)

9.3 Remediation of Mycotoxins From Food and Agriculture Using Nanotechnology

9.3.1 Removal of Mycotoxins by Using Adsorption

Traditional enzyme-based decontaminant methods for the removal of mycotoxins from food have several drawbacks, such that the toxin may remain in the food matrix or that it can also result in highly toxic secondary metabolites (Jard et al. 2011; Manubolu et al. 2018). Although nanotechnology has advanced substantially in the detection of mycotoxins, very few studies are available on the elimination or removal of mycotoxins from various food matrices. Among the various immobilization approaches, magnetic nanoparticles are the best solid carriers due to their exclusive high saturation magnetization for facile separation and reusability (Ansari and Husain 2012). Some of the recent applications for removal of mycotoxins from various biological matrices are described in this section (Table 9.5).

Surface active maghemite nanoparticles (SAMNS Fe_2O_3, 11 ± 2 nm) were used for the removal of citrinin in Monascus treated foods, which can be used in the food industry (Magro et al. 2016). The SAMN@citrinin conjugate complex was thoroughly characterized structurally and was magnetically isolated. The offered magnetic separation system was based on the citrinin binding on SAMNS surface and is based on the firm iron chelating keto-enol group on the toxin. In another study, Luo

Table 9.5 Degradation or removal of mycotoxins

Mycotoxin	Nanomaterial	Method	Max. degradation or recovery (%)	Sample matrix	Refs.
Applications for removal					
Citrinin	Surface active maghemite nanoparticles (Fe_2O_3)	Magnetic separation	70% removal	Biological matrixes	Magro et al. (2016)
Patulin	Chitosan-coated Fe_3O_4 nanoparticles	Magnetic adsorbent	99% within 60 min	Fruit juice; water or others	Luo et al. (2017)
Patulin	Magnetic chitosan-Fe_3O_4 nanoparticles coated with *Candida utilis*	Magnetic adsorbent	90%	Orange juice	Ge et al. (2017)
Patulin	Zirconium-based MOF UiO-66(NH_2) @Au-Cys	Adsorption	4.38μg mg^{-1}	Apple juice	Liu et al. (2019)
Patulin	Magnetic multi-walled carbon nanotubes (MWCNT)	Magnetic adsorbent	640.2μg g^{-1}	Aqueous systems	Zhang et al. (2019)
ZEA; fumonisin B1 and B2; DON	Magnetic graphene oxide nanocomposites	Adsorption	37–69%	Animal feed: palm kernel cake	Pirouz et al. (2017)
Applications for degradation					
DON	Graphene/ZnO hybrids	Photocatalytic (UV light)	99% of DON (15 ppm) within 30 min	Aqueous suspension	Bai et al. (2017)
AFB1	Scandium doped $SrTi_{0.7}Fe_{0.3}O_3$	Photocatalytic (visible light)	88% at pH 8 within 120 min	Water	Jamil et al. (2017)
AFB1	CdS/WO_3 composites	Photocatalytic (visible light)	80%	Water	Mao et al. (2019)
AFB1; AFB2	TiO_2, immobilized on a glass support	Photocatalytic (high-pressure UV–vis irradiation)	99% with 4 min	Peanut oil	Magzoub et al. (2019)
AFB1	TiO_2 (P25, Degussa) layer in a closed-loop reactor	Photocatalytic (UV light)	60%	Peanut oil	Xu et al. (2019)
AFB1	Activated carbon supported TiO_2 composites	Photocatalytic (visible light)	98% bare TiO_2 76%	Water	Sun et al. (2019)
DON	Dendritic α-Fe_2O_3	Photocatalytic (visible light)	90%	Aqueous solution	Wang et al. (2019)

et al. (2017) reported the synthesis of chitosan coated Fe_3O_4 nanoparticles as a magnetic adsorbent for the patulin removal from fruit juice. Further, magnetic chitosan Fe_3O_4 nanoparticles coated with deactivated *Candida utilis* cells and used as a new biosorbent for patulin removal from fruit juice (Ge et al. 2017). Similarly, for patulin removal, magnetic multi-walled carbon nanotube (MWCNT) adsorbent was fabricated, which can be regenerated using NaOH solution for recycling usage up to four cycles (Zhang et al. 2019).

9.3.2 Degradation of Mycotoxins Using Nanotechnology

Recently, there has been a growing interest in photocatalytic degradation of many organic pollutants, including mycotoxins. Photocatalytic degradation has numerous advantages, such as being environmentally friendly, low–cost, and requiring mild conditions (Bhatkhande et al. 2002). Various photocatalysts, both under the UV light and visible light irradiation, are presented in Table 9.5. Nevertheless, as UV light accounts for only 4% of the total sunlight, visible light photocatalysts are more beneficial for practical degradation of organic pollutants in natural systems (Pathakoti et al. 2013, 2018).

Bai et al. (2017) reported the synthesis of graphene/ZnO hybrids using a single one-step hydrothermal method for the photodegradation of DON under UV irradiation. As shown in Scheme 1a, the proposed photodegradation mechanism is due to the formation of superoxide radicals at the conduction band. It is recognized that the graphene increases the adsorption capability and the ZnO increases the photocatalytic achievement by graphene hybridization (Fig. 9.6). Additionally, the degradation products of DON were analyzed by using ESI/MS analysis.

Recently, Mao et al. (2019) reported the photocatalytic inactivation of AFB1 at hypertoxic site C8=C9 under visible light treatment using the Z-schematic composite, clew-like WO_3 adorned with CdS nanoparticles, synthesized by the microwave supported hydrothermal and precipitation procedure (Fig. 9.7). The reaction of hydroxyl radical formation with C8=C9 bond of AF for the photocatalytic inactivation was confirmed by radical trapping experiments and ^{18}O isotope labeling. Further, the degraded products were identified by using High Resolution Mass Spectroscopy, and the theoretical calculations using the density functional theory were used to confirm the formation of hydroxyl radicals at the C9 site, thereby forming AFB1-9-hydroxy.

Similarly, another study reported the 10 mole% Scandanium doped $SrTi_{0.7}Fe_{0.3}O_3$ (band gap, 1.58 eV) was synthesized for the photodegradation of afltoxinB1 under visible light irradiation (Jamil et al. 2017). Further, the degraded products were identified using gas chromatography-mass spectrometry (GC-MS), which are nontoxic to *Vibrio fischeri* organism. Hence, it is safe for water treatment, and moreover, both the catalyst and treated water can be reused for up to eight times. The mechanism of oxidation is due to rupture of short chain aliphatic alcohol, which leads to total degradation.

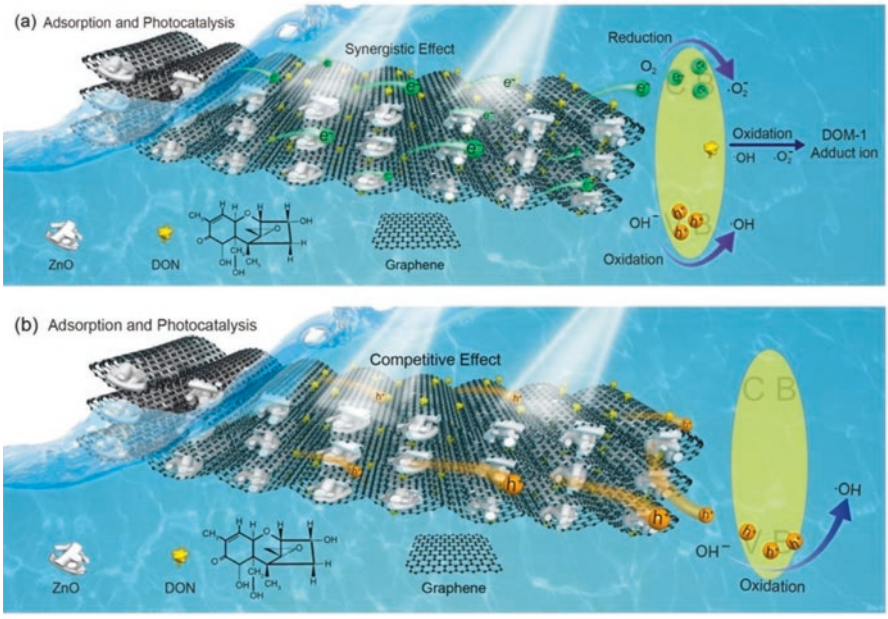


Fig. 9.6 Schematic drawing illustrating synthetic route and the mechanism of charge separation and adsorption-photocatalytic process over graphene/ZnO hybrid photocatalysts under UV light irradiation. Obtained with permission from Elsevier, Ref: (Bai et al. 2017)

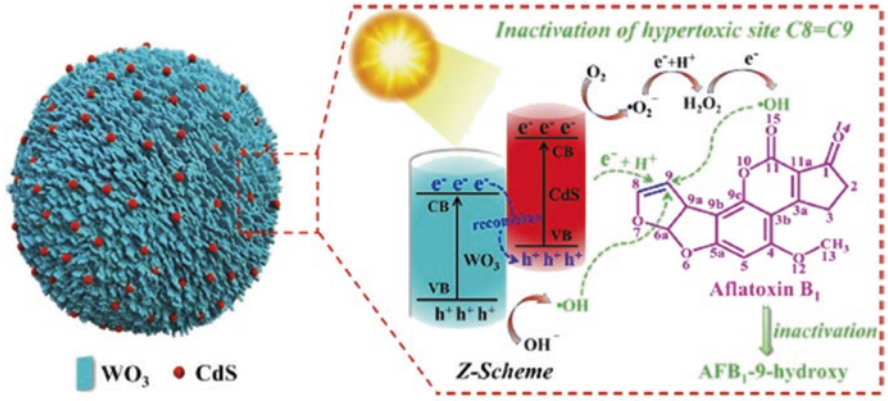


Fig. 9.7 Illustration of the proposed mechanism over CdS/WO_3 composites for inactivating the hypertoxic site [(C8=C9) in AFB1 under visible light irradiation. Obtained with permission from Elsevier, Ref. (Mao et al. 2019)

A study by Magzoub et al. (2019) reported the photocatalytic detoxification of AFB1 and AFB2 from its real sample matrix, peanut oil, using the TiO_2 that was immobilized on a glass support under high pressure UV–vis irradiation. It is noteworthy that the TiO_2 treatment did not alter the physicochemical parameters of the

oil, such as iodine value, peroxide value, saponification value, fatty acid composition, and free fatty acids. Furthermore, the photocatalyst can be recycled up to ten cycles without reducing its effectiveness. The proposed mechanism is the formation of hydroxyl radicals and is comparable to previous reports (Jamil et al. 2017; Mao et al. 2019).

Likewise, Xu et al. (2019) reported the closed loop photocatalytic reactor comprising a glass tube holding TiO_2 catalyst for the decontamination of AFB1 from peanut oil. Based on their experimental data on AF detoxification, a theoretical model, a Weibull distribution model, has been proposed, which offers a good depiction for the photocatalytic process kinetics.

A simple hydrothermal method was described by Sun et al. (2019) for the photocatalytic degradation of AFB1 using an activated charcoal/TiO_2 composite under UV–vis irradiation. This newly synthesized composite had higher degradation efficiency when compared to the bare TiO_2 and the catalyst can be regenerated and recycled up to four cycles with 80% degradation efficiency. Formation of hydroxyl radicals plays a vital role in the degradation of AF, whereas superoxide radicals do not have an effect.

Another effective approach is the improvement of the photocatalyst, dendritic-like α-Fe_2O_3, for the DON degradation under visible light irradiation (Wang et al. 2019). A simple hydrothermal has been proposed for dendritic-like α-Fe_2O_3, which has better photocatalytic activity (more than 90% in 2 h) than the commercial α-Fe_2O_3. After degradation the intermediate products formed were identified by high perrformance liquid chromatography-mass spectrophotometry (HPLC-MS) and the major toxicity group, epoxy group at C12 and C13 and hydroxyl groups are destroyed in DON, thereby providing an efficient and green technology for mycotoxin decontamination.

9.4 Summary and Conclusion

The occurrence of mycotoxins in the food chain is a major safety concern around the world. Control of mycotoxins commonly depends on the suitable care taken during pre- and post-harvest conditions. The potential and present uses of nanotechnology in farming and food industries offer various significant benefits to ensure the microbial food quality and safe of nourishment items. The use of gold, QDs, and magnetic nanoparticles has significantly enhanced the biosensor technology for the mycotoxin analyses. All the above discussed nano-based sensors developed for detection and sensing of mycotoxins can be easily applied in the fields and also can be operated by unskilled personnel. Further, the sensitive and early detection of mycotoxins in food will be beneficial in eliminating these toxins to enter into food chain and thereby protects human health. Although QDs have various advantages due to their optical properties in comparison to other nanoparticles in sensing applications, but they are not cost-effective and expensive till date. In order to overcome the overestimation, false positive or underestimation, usage of more than one

technique such as multiple sensor devices, which can be facile to fulfill the rapid monitoring and control of mycotoxins is essential.

Acknowledgments The author (K.P.) is thankful to the National Science Foundation (NSF/CREST HRD-1547754) for financial support.

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