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Nanoplasmonic biochips for rapid label-free detection of imidacloprid pesticides with a smartphone

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ABSTRACT

The widespread and intensive use of neonicotinoid insecticides induces negative cascading effects on ecosystems. It is desirable to develop a portable sensitive sensing platform for on-site screening of high-risk pesticides. We combined an indirect competitive immunoassay, highly sensitive surface plasmon resonance (SPR) biochip and a simple portable imaging setup for label-free detection of imidacloprid pesticides. The SPR biochip consists of several capped nanoslit arrays with different periods which form a spectral image on the chip. The qualitative and semiquantitative analyses of pesticides can be directly observed from the spot shift on the chip. The precise semiquantitative analyses can be further completed by using image processing in a smartphone. We demonstrate simultaneous detection of four different concentrations of imidacloprid pesticides. The visual detection limit is about 1 ppb, which is well below the maximum residue concentration permitted by law (20 ppb). Compared to the one-step strip assay, the proposed chip is capable of performing semiquantitative analyses and multiple detection. Compared to the enzyme-linked immunosorbent assay, our method is label-free and requires simple washing steps and short reaction time. In addition, the label-free chip has a comparable sensitivity but wider working range than those labeling techniques.

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1. Introduction

To increase the efficiency of farming, a family of compounds called neonicotinoids for soil injection, seed treatments and foliar spray is commonly used. Recent studies have shown that widespread and intensive use of neonicotinoid insecticides has been linked to declining honey bee and bird populations and negative cascading effects on ecosystems (Henry et al., 2012; Whitehorn et al., 2012; Hallmann et al., 2014; Goulson, 2013; Van der Sluijs et al., 2015). Imidacloprid is the most widespread insecticide among the neonicotinoids. It kills insects by binding nicotinic acetylcholine receptors (nAChR) in the central nervous system of insects. Imidacloprid is highly specific for the nAChR found in insects and less harmful to mammals. However, it is soluble in water and has a long lifetime greater than 1000 days, which make it accumulate in soils or run off into water sources and ultimate spread through the environment. Therefore, an effective and quick detection of such high-risk pesticide is important to prevent potential negative effects on ecosystems. For efficient pesticide identification and quantification, standard analytical methods such as high performance liquid chromatography (HPLC), gas chromatography (GC) and liquid chromatography (LC) combined mass spectrometry (MS) are commonly used. Although being sensitive and specific, these methods are high cost, time-consuming sample-preparation procedures and need highly trained technical personnel. In order to fulfill the requirement of rapid, simple, selective and sensitive detection, enzyme-linked immunosorbent assays (ELISAs) have been developed for detection of neonicotinoids in different matrices (Li and Li, 2000; Kim et al., 2004, 2006; Watanabe et al., 2004, 2013; Fang et al., 2011; Wang et al., 2012). However, repeated washing steps make the ELISA method difficult for one-step multiple assays. In contrast to the

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ELISA method, an immune-chromatographic assay or lateral-flow assay using colloidal gold as the label requires an assay procedure of only one step (Posthuma-Trumpie et al., 2009). It is portable, easy and convenient to use without specialized laboratory equipment. Such a strip assay has been utilized for qualitative detection of imidacloprid through naked eyes (Xu et al., 2012). Different from the previous techniques requiring the assistance of labels, surface plasmon resonance (SPR) sensing is a real-time and label-free detection technique which has been employed for detecting imidacloprid (Homola et al., 1999; Dinga et al., 2012; Ding and Yang, 2013). Commercial sensing platforms utilize an optical prism to induce propagating surface plasmon polaritons in thin noble films and enable real-time and label-free measurements of biomolecular binding affinity. In addition to the prism coupling method, metallic nanostructures offer a simpler way for SPR excitation (Raether, 1988; Ebbesen et al., 1998; Stewart et al., 2008; Anker et al., 2008). Periodic metallic nanohole, nanoslit or nanogrid arrays have been utilized for biosensing applications (Brolo et al., 2004; Gordon et al., 2008; Im et al., 2011; Brolo, 2012; Lee et al., 2007, 2012a, 2012b). However, the majority of SPR sensing is performed on dedicated and expensive instruments. In this study, we combined an indirect competitive immunoassay, capped-nanoslit-based SPR biochip and simple portable imaging system for label-free detection of imidacloprid pesticides. The sensing chip, similar to a tiny spectral analyzer, consists of several capped nanoslit arrays with different periods. The distribution of the transmitted light from these arrays comprises a spectral image on the chip. The detection system is a smartphone with a cheap light-emitting diode (LED) and a narrowband filter. When the analyte is on the surface of the chip, the brightest spot of the spectral image is shifted. The qualitative and semiquantitative analyses of the analyte can be conducted by observing the spot shift on the chip through naked eyes or smartphones. We demonstrated simultaneous detection of different concentrations of imidacloprid pesticides. The visual detection limit is about 1 ppb, which is below the maximum residue concentration permitted by law (20 ppb). The lowest detectable concentration for the imidacloprid pesticide can be further improved by using image processing in the smartphone. Such a portable sensing platform takes advantages of multiple detection, semiquantitative and qualitative analyses, rapid determinations, user-friendliness and low cost. It can be utilized for on-site screening of pesticides and considered as a cost-effective complement technique to the chromatography methods.

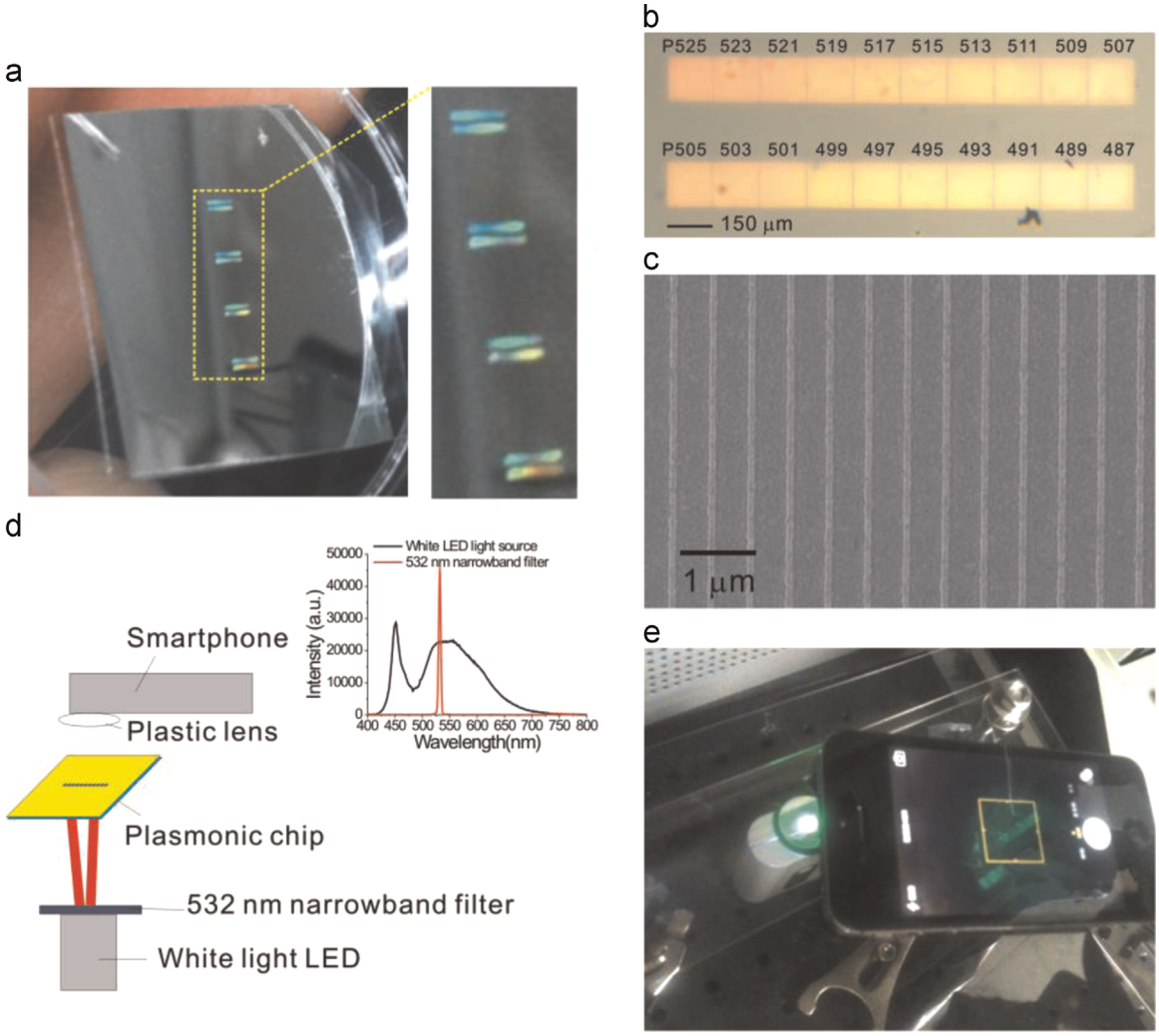

Fig. 1. Fabrication of plasmonic biochips and optical setup. (a) Optical image of the plasmonic biochip. There were four sets of capped nanoslit arrays on the chip. (b) The transmitted light image of the biochip using white light. The period of the nanostructure ranged from 487 to 525 nm. (c) The scanning electron microscope image of a capped nanoslit array with 516 nm period. (d) Schematic configuration of the portable optical system for the image measurement. The system composed of a smartphone, LED light source and narrowband filter. (e) Optical image of the detection system using a smartphone.

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