

Succinyl-NCQD Enhanced LSPR for Chlorine Detection in Lipid Matrices

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Abstract

Chlorine contamination in crude palm oil (CPO) is a key factor in the formation of 3-monochloropropane-1,2-diol (3-MCPD), a harmful contaminant with carcinogenic potential. Existing detection methods such as ion chromatography (IC) and total chlorine analyzers (TCA) are accurate but complex, time-consuming, and not suitable for rapid or on-site testing. This study was conducted to develop a localized surface plasmon resonance (LSPR) optical sensor for detecting organic chlorine in edible oil matrices. The main objectives of this study are to investigate LSPR sensing for chlorine detection, fabricate a composite-based LSPR sensor using succinyl-functionalized nanocarbon quantum dots (succinyl-NCQD) and silver nanotriangles (AgNT), and evaluate its sensitivity in detecting low chlorine concentrations. Succinyl-NCQD was chosen as the sensing material to improve binding interaction and signal response. The sensor showed a strong linear response in the 0.5-5 ppm range with a sensitivity of $1.6877 \text{ nm ppm}^{-1}$, a limit of detection (LOD) of 0.56 ppm, and a limit of quantification (LOQ) of 1.85 ppm.

1. Introduction

Recent discoveries of contaminants in lipid-based mediums, such as 3-monochloropropane-1,2-diol (3-MCPD) esters, have raised concerns about oil quality and safety due to their potential cancer risks [1]. 3-MCPD is a byproduct formed during high-heat oil refining, especially in the deodorization stage of palm oil processing, where temperatures above 225°C and hydrogen chloride (HCl) are used [2]. This process, which also involves acylglycerols, chloride content, and processing time, can lead to the formation of 3-MCPD esters, a known carcinogen [3]. Palm oil, in particular, has been shown to contain higher levels of 3-MCPD compared to other fats, making its safety a global concern.

Regulatory authorities have established two distinct maximum permissible levels for 3-MCPD in vegetable oils and fats intended for consumer use or as food ingredients. The first category encompasses unrefined oils, refined oils and fats derived from coconut, maize, rapeseed, olives (excluding olive pomace oil), sunflower, soybean, palm kernel, and their respective mixtures, with a maximum allowable limit of 1.25 ppm. The second category includes other refined vegetable oils, fish oils, oils from marine organisms, and mixtures within this classification, which are subject to a higher maximum limit of 2.5 ppm. The need for reliable, efficient methods to monitor these contaminants is critical. Traditional methods like gas chromatography-mass spectrometry (GC-MS) and liquid chromatography-tandem MS (LC-MS/MS) are highly sensitive but require complex sample preparation and instrumentation. This has led to the development of more user-friendly alternatives, such as optical biosensors, which offer direct, label-free detection with minimal sample preparation and faster analysis.



Techniques like surface plasmon resonance (SPR) and surface-enhanced Raman spectroscopy (SERS) are gaining attention for their high sensitivity, reproducibility, and ease of use.

2. Materials and Methodology

Succinyl-NCQDs were synthesized hydrothermally, and AgNTs were prepared by chemical reduction [4]. Glass slides were cleaned with piranha solution and functionalized with APTES. A 1:4 AgNT-succinyl-NCQD mixture in PVA was drop-cast (90 μ L) onto slides and dried at 40 $^{\circ}$ C for 10 minutes. LSPR spectra as shown in Figure 1 were recorded using an Ocean Optics HR4000CG-UV-NIR spectrometer (350-850 nm) after immersing samples in lipid matrices containing range of organic chlorine from 0.5 ppm to 50 ppm.

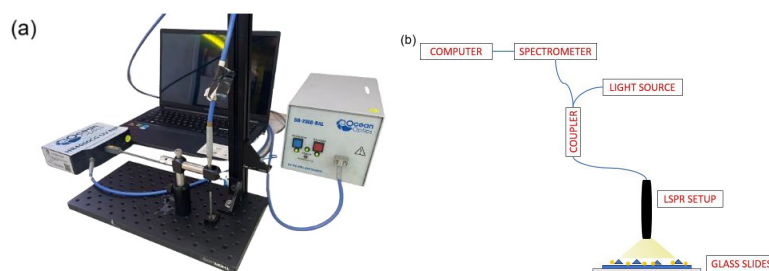


Figure 1. LSPR setup of chlorine detection a) Real setup, b) Schematic diagram.

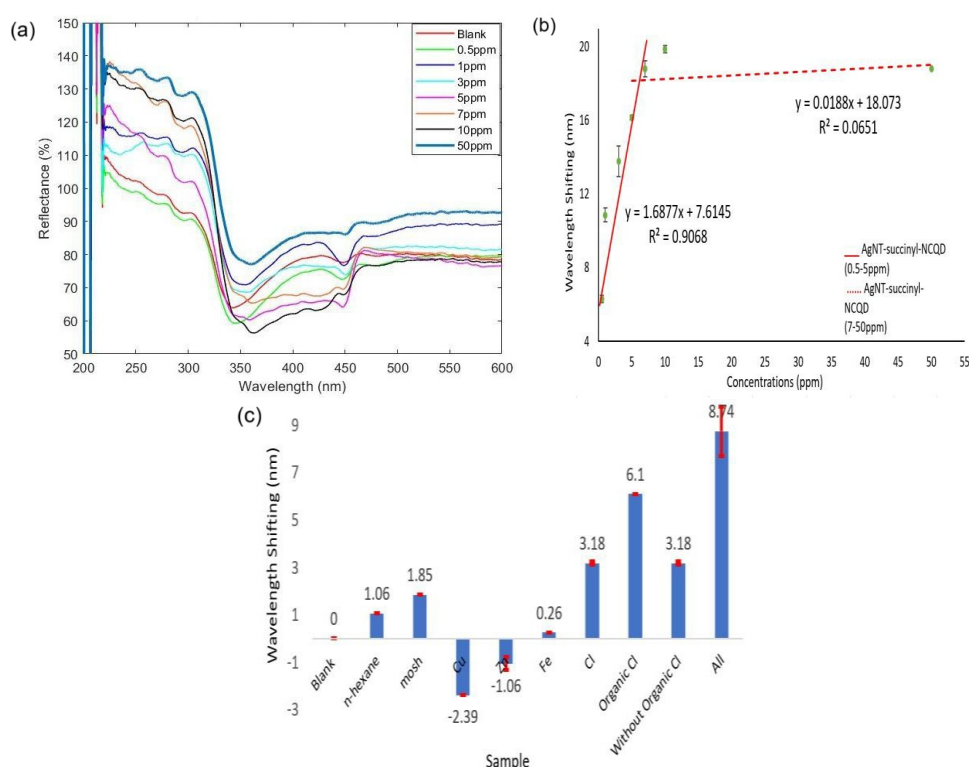


Figure 2. (a) LSPR spectra of AgNT-succinyl-NCQD sensor with chloride concentrations from 0.5 to 50 ppm, showing increasing wavelength shifts, (b) Calibration curve showing strong linearity from 0.5-5 ppm and saturation beyond 7 ppm, (c) Selectivity test confirming high response to chloride and organic chlorine, with minimal interference from other substances.

3. Result and Discussion

The AgNT-succinyl-NCQD sensor exhibited strong sensitivity and linearity for chlorine detection in the low concentration range of 0.5-5 ppm. The calibration curve followed the regression equation $y = 1.6877x + 7.6145$,



with a high correlation coefficient ($R^2 = 0.9068$), indicating reliable performance for trace detection. The calculated LOD was 0.56 ppm, and LOQ was 1.85 ppm, confirming the sensor's capability for accurate low-level chlorine monitoring. However, in the higher range of 7-50 ppm, the response dropped significantly.

The curve flattened, with a poor correlation ($R^2 = 0.0651$) and a sensitivity of just $0.0188 \text{ nm ppm}^{-1}$. This decline is attributed to saturation of the sensor surface, where active binding sites on the succinyl-NCQD become fully occupied, resulting in limited LSPR signal change beyond $\sim 10 \text{ ppm}$ [5]. Selectivity tests supported the sensor's specificity. Notable peak shifts were observed for chloride ions ($\sim 3.18 \text{ nm}$), organic chlorine ($\sim 6.1 \text{ nm}$), and their combination ($\sim 8.74 \text{ nm}$), while minimal or negative shifts occurred for n-hexane, MOSH, and metal ions like Cu^{2+} and Fe^{2+} . This confirms the sensor's strong preference for chlorine species and minimal interference, making it highly suitable for detecting organic chlorine in complex matrices such as crude palm oil. Figure 2 shows the result for the sensor's performance for chlorine detection in lipid-based medium.

4. Conclusion

The LSPR sensor based on AgNT and succinyl-NCQD demonstrated high sensitivity and selectivity for detecting organic chlorine in crude palm oil. Its rapid response and low detection limit make it a promising tool for on-site monitoring of chlorine contaminants.

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