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Fluorescent Method for Determination of residue toxic Insecticide Abamectin Using PbS Quantum Dots-Based Gelatin in Water Samples

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ABSTRACT

In this study the residue behavior of toxic insecticide abamectin on surface water with fluorescent chemical sensors because they are non-destructive, the ability to show decomposed concentrations, fast response, and high was investigated. In this research, a chemical sensor was synthesized PbS functionalized with gelatin quantum dots for toxic abamectin. The calibration curve was linear in the range of (0.05 to 10.0 μgL^{-1}). The standard deviation of less than (1.1 %), and detection limits of the method (0.05 μgL^{-1}) in time 60 s, 350 nm were obtained for sensor level response PbS Quantum Dot–Gelatin nanocomposites sensor with (95%), confidence evaluated. The observed outcomes confirmed the suitability recovery and a very low detection limit for measuring the toxic abamectin. The chemical PbS Quantum Dot–Gelatin nanocomposites sensor as excellent sensor in the practical application of toxic abamectin related to residue management is in Water Samples. The method fluorometric can be used as to estimate the appropriate PHIs and can also support authorization of plant protection products as supplementary information.

Keywords: Abamectin, Insecticide, Fluorescence, PbS with Gelatin nanocomposite, Quantum Dot

1. INTRODUCTION

Abamectin is a type of macrocyclic lactone that is produced from a soil bacterium called Streptomyces avermytilis. And it is used as an insecticide and acaridae in plants and animals [1]. This insecticide belongs to the avermectin family and is a mixture of avermectin B1a (minimum 80%) and B1b (maximum 20%). This insecticide should be kept at a temperature of 2-7 degrees Celsius. Abamectin insecticide attacks the nervous system of insects and ticks and paralyzes them within a few hours. And it is not possible to return to the original state and it is activated as soon as it enters the stomach. Table 4-2 shows the toxicity of

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this insecticide in different species of animals [2,3]. Assuming that they will be applies according to the authorized agricultural patterns of good agricultural practices. On the other hand, the misuse of pesticides may lead to extensive concentrations of residues in the agricultural products, which has forced international agencies and governments to established maximum residue limits (MRLs) as to ensure that safe, to the consumer, products enter the market [4,5].

Since toxic abamectin is the widely applied an acaridae, monitoring and determination of toxic abamectin in ground and surface waters and in cultivated areas where it is used are of high significance. Until now, different methods such as gas chromatography [6], liquid mass chromatography [7], solid-phase extraction and liquid chromatography [8], liquid chromatography–electrospray ionization–tandem [9] Molecularly imprinted polymers (MIPs) [10,11]. Despite the selectivity and specificity of some analytical techniques, they are time-consuming and require a larger amount of samples. On the other hand, fluorometrically techniques have many benefits in comparison to the others, for example, simplicity, low-cost, accurateness, sensitivity, ability to determine, can be the good candidate in studies on the persistence of insecticide in water samples [12-14].

In recent years, fluorescent sensors have attracted much attention in the detection of herbicides due to their excellent properties of easy operation, high sensitivity, selectivity, and real-time monitoring [15-17]. In the past few decades, a great variety of fluorescence probes have been reported for the determination of herbicides including organic dyes [18,19] and quantum dots (QDs) [20]. Those QDs, Therefore, the necessity of developing alternative and ecologically friendly materials became apparent [21]. Also as method for the detection of various analytes and their characterization has become a pivotal research area in materials science research. However, PbS nano-composites can by various methods such as the application of stabilizing and reducing chemicals of glutaraldehyde, reduction prepared [16,17]. Gelatin as a natural, completely non-toxic, and biocompatible polymer derived from collagen, is a very suitable option for coating nanoparticles of lead sulfide quantum dots as it is shown in (Fig. 1). Because it can be made into fine and stable particles and at the same time it can be used as a transfer agent which creates significant stability in the form of cross-linking in these materials, and more importantly, its by-products are absorbable and to be destroyed or decomposed [22,23].

Quantum dots PbS–gelatin nanocomposites provides several advantages such as simple, fast and clean synthesis without the use of toxic and dangerous compounds as a highly stable and reusable ecofriendly catalyst, more nanocomposites quantum dots can be molded into a variety of different forms, including sheets and three-dimensional arrays, in contrast to many other semiconducting materials, more can also be easily combined with organic polymers, dyes or porous films. In the colloidal form, or dissolved in a solution, quantum dots can be processed sheets [24,25]. Despite all these valuable properties, their intrinsic instability and the associated degradation is one of the important challenges for large scale production, thus restricting their applicability. However, the fascinating optical characteristics of quantum dots (QDs) such as light absorption, and photoluminescence make them alluring candidates for materials measurement when against other commercial [12,26].

In the present work, designed the successful of nano-probe for determining toxic abamectin a fluorometric method in different water samples. In this research, for measure toxic abamectin various effective factors such as (pH, abamectin) concentration, PbS quantum dot–gelatin nanocomposite concentration, time, etc.). On the response of the method and obtaining the optimal test values and obtaining the linear range, detection and accuracy of the method presented in the measurement of insecticide toxic abamectin by a fluorometric new method in real samples was checked. The method by fluorescence emission intensity introduced to measure toxic abamectin in water samples and can be used for other samples.

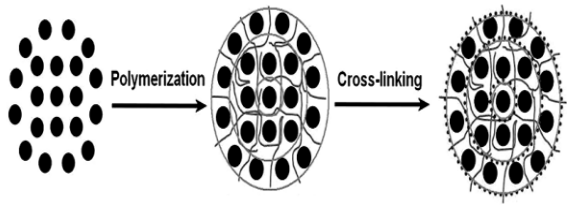

Fig. 1. Schematic illustration of the PbS quantum dots sensor functionalized with gelatin synthesis.

2. EXPERIMENTAL

2.1. Reagents and equipment

All chemicals of lead nitrate $Pb(NO_3)_2$ (99%), sodium sulfide (Na_2S) (99.0 %), gelatin, and glutaraldehyde were bought from Merck Company. A stock solution of 1000 mg/L of analytical grade abamectin (99.0%) from Sigma-Aldrich, was prepared by dissolving 0.1 g of the toxic diphenylamine in solution and diluting it to 100 mL in a volumetric flask.

All the recordings were done at room temperature, measurements toxic abamectin were done using a Horiba JY Fluorolog-3 molecule fluorometer (Paris Company, France). Time-resolved luminescence intensity decay was registered, and by using a 330 nm laser light source, samples were excited. X-ray diffraction from the device Rigaku-Dmax 2000 used. X-ray diffraction test pattern analysis by JCPDS (No. 5-592), software was performed. Fourier transform infrared (FT-IR) spectra were registered on a PerkinElmer (FT-IR spectrum BX, Germany).

2.2. Pretreatment of water samples

A water sample from Fahlyan River warer Fars, Karon River warerAhvaz, Karkheh dam water, (polluted dams and river water with a high content of organic components) was collected in 2.5 L brown glass bottles. Immediately after arrival in the laboratory, the samples were filtered through 1 mm glass fiber filters and 0.45 mm cellulose acetate filters, sequentially, to remove suspended particles. After standing for 24 h in the refrigerator, the samples were filtered by a piece of filter paper) where the toxic abamectin content in each sample was determined blue at optimum conditions. Each test was repeated at least three times for consistency of the results. Abamectin content of different water samples and their recovered counterparts were subjected to further investigation. The samples were then adjusted to pH 4.0 and immediately analyzed by [12,13].

2.3. Synthesis of PbS Quantum Dot–Gelatin nanocomposites

The nanoparticle PbS was synthesized in reactive solution prepared using zinc nitrate $Pb(NO_3)_2$ and sulfide sodium (Na_2S) with concentration of (0.1 M and 0.1 M). The Gelatin pellets were used as a base medium and its concentration was set to (0.1 M). 20 mL of all the above solutions were prepared separately, using distilled water as a solvent and mixed together in a beaker. 2 ml of glutaraldehyde (25%) was added into the solution as a complexing agent, which can

easily bind the metal ions. The reactive vessel with solution was immersed into 20 ml acetone maintained at 40°C and pressure of 1×10^{-5} mbar. The powder was collected and dried in a hot air oven [13,27].

2.4. Procedure fluorescent detection measurements

In this procedure, PbS Quantum Dot–Gelatin nano composites (2.5×10^{-2}M), then 2 ml of glutaraldehyde (25%), 1 mL of Robinson buffer (pH 5.0), and different concentrations of toxic abamectin (10.0 μgL^{-1}) were added to 10 mL volumetric flasks and diluted with double distilled water. The difference between the quantities of the increasing fluorescence emission in a wavelength equal to (350 nm) in a time interval equal to (30-60 s), was estimated [12-14]. By adding toxic abamectin to the solution, it was observed that fluorescence emission intensity of the acetonitrile solution of PbS quantum dot–gelatin nanocomposites at wavelength of (350 nm) dropped. All reaction steps were repeated by increasing the concentration ($2.0\ \mu gL^{-1}$) of the toxic abamectin every (10 s). Moreover, all the steps were repeated for a reaction. In the fluorometric of toxic abamectin (ΔI b) and ultimately (ΔI) I_0 blank-I sample. There was a sharp change in the fluorescence emission of the sensor in the 350 nm region, a continuous increase of toxic abamectin at intervals of (10 s) in solution and changes in the fluorescence emission intensity of the sensor, peak fluorescence emission during 350 nm, with an increase in fluorescence emission intensity, can be seen in (Fig. 2a). UV–visible spectrum (AAb). All these steps would be repeated for a reaction without the presence of toxic abamrctin (AAb), finally (AA) AAblank-AAsample is calculated. The spectrum changes are due to the addition of toxic abamectin in the range of ($0.02\ \mu gL^{-1}$ at ۱۲.0 μgL^{-1}) and the formation of a complex. As can be seen, the complex (toxic Abamectin sensor) has two absorption peaks, the first at a wavelength of ۳۵۰ nm and the second at a peak Appears at a wavelength of 425 nm. (Fig. 2b) [16,28].

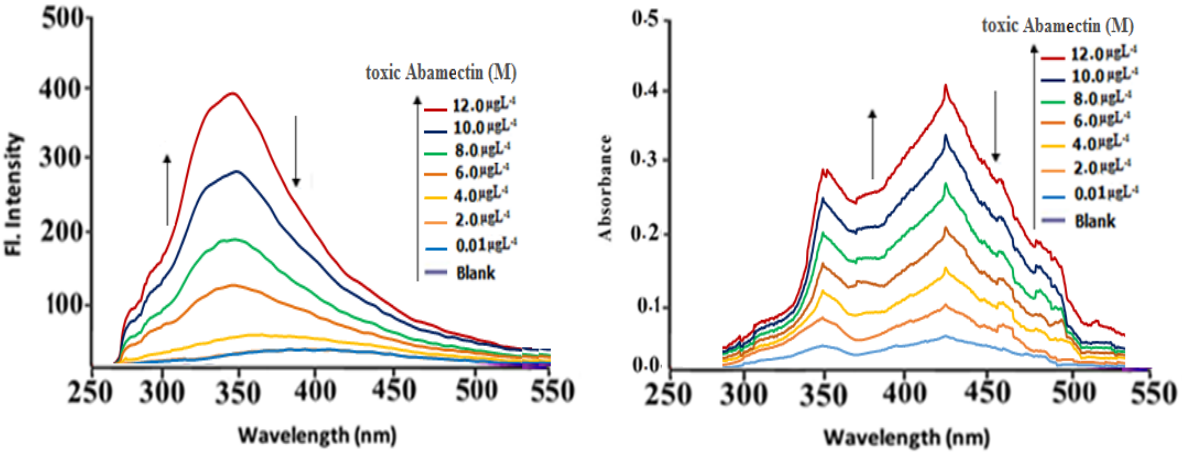

Figure 2. (a) The Fluorescent detection of toxic abamectin by PbS Quantum Dot–Gelatin nanocomposites in 350 nm and added solution increasing of the toxic abamectin ($2.0\ \mu gL^{-1}$) in time 10 s, in Robinson buffer solution. (b) The absorption spectra of toxic abamectin by PbS Quantum Dot–Gelatin nanocomposites added solution increasing of the toxic abamectin solution ($10.0\ \mu gL^{-1}$) in time 10 s, in Robinson buffer solution.

3. RESULTS AND DISCUSSION

3.1. Characterization

The results of IR analysis of the samples in the presence of uncoated PbS quantum dots and the quantum dots coated with gelatin can be seen in (Fig. 3a). A broad absorption band in the region of 3445 cm^{-1} is indicated by the presence of OH-hydroxyl groups in the sample. The absorption bands in region 3416 cm^{-1} belong to the N–H and the vibrations in region 1733 cm^{-1} belong to the C=O group. Absorption bands in the area of 1350 and 1380 are related to nitro compounds. And 985 cm^{-1} and 1110 cm^{-1}, respectively, prove the Pb–S covalent bond in the substrate of the PbS Quantum Dot–Gelatin nanocomposites substrate, respectively [16,29]. The XRD spectra of the PbS QDs –Gelatin nanocomposites substrate are shown in (Fig. 3b). Specific peaks for nanocomposites to the great intensity of signal at 25.8 (111) and 30.5 (200) confirmed that there has been a slight amount of material in amorphous state. XRD pattern of crystal, JCPDS (No. 5-592) [30]. As shown in (Fig. 3c), which relates to differential thermal analysis (DTA) of PbS QDs –gelatin nanocomposites, at 200 and 350°C, bands related to the decomposition of gelatin chains by weight are seen. Molecular is low. Also, at 530°C, a large calorific band is observed due to the large amount of gelatin and organic compounds remaining in the sample, which are destroyed at temperatures above 600°C. The results indicate that lead sulfide particles have a suitable coating of the desired polymer. As shown in (Fig. 3d), which deals with the thermal gravimetric analysis (TGA) of PbS QDs –gelatin nanocomposites, at temperatures below 600°C for gelatin we see a 95% weight reduction. While under the same conditions for PbS QDs with a high molecular weight polymer, we see only 32% weight loss [30,31].

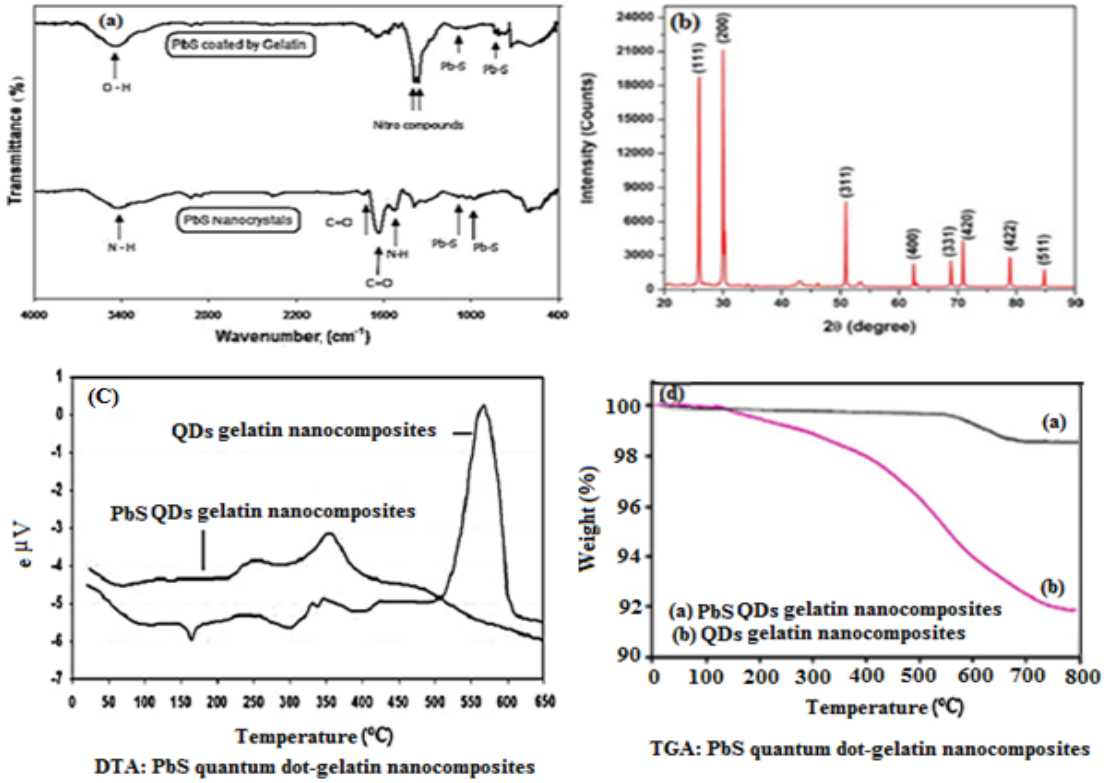

Figure The (a) FT-IR (b) XRD (c) DTA (d) TGA of PbS QDs–Gelatin nanocomposite.

3.2. Optimization of Sensing Conditions

Obtaining an exceptionally sensitive response in detecting toxic abamectin rests upon the systematic optimization of pH, PbS QDs–Gelatin nanocomposites and incubation time.

3.2.1. Effect of pH

After measuring the fluorescence intensity of the solution, a thorough investigation was carried out on the influence of pH value in the range of 2.0–9.0 for the toxic abamectin–PbS QDs – Gelatin nanocomposite complex at 350 nm. This phenomenon might be because of the weak complexion at lower pH values (pH < 4.0). On the other hand, the reduced response of the proposed PbS QDs – Gelatin nanocomposite sensor for toxic abamectin determination at pH > 4.0 could be due to a possible formation of hydroxide of toxic abamectin in solution. Thus, pH 4.0 was selected as favorable pH for all subsequent experiments (Fig. 5) [16,28].

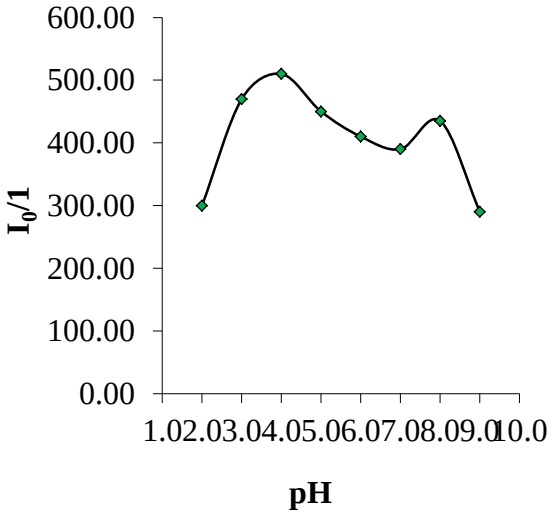

Figure. 5. Effect of pH in the fluorescence intensity rate, PbS QDs–Gelatin nanocomposite ۲،۵×۱۰-۲M, glutaraldehyde (25%), time 60 s, 350 nm) in Robinson buffer solution.

3.2.2. Effect of PbS QDs–Gelatin nanocomposite

The Effect of the PbS QDs–Gelatin nanocomposite, 1 mL of 10 µg L^{-1} toxic abamectin solution, 2 mL of 25% glutaraldehyde, and 1 mL of PbS QDs–Gelatin nanocomposite (0.5×10^{-2}M to 4.0×10^{-2}M) were mixed in a 10 mL volumetric flask using distilled water to establish the impact of PbS QDs–Gelatin nanocomposite concentration on the reaction rate. Again, the fluorescence intensity of the solution was assessed. The previously mentioned operation was replicated for a blank solution (the solution not containing toxic abamectin). The findings are exhibited in (Fig. 6). Based on the results, the concentration of 2.5×10^{-2} M was determined to be optimum [12,17].

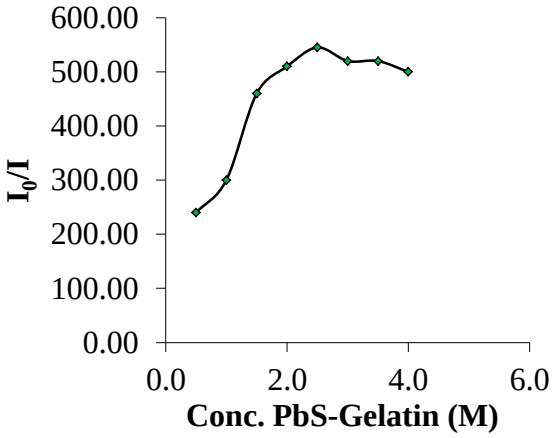

Figure. 6. Effect of PbS QDs–Gelatin nanocomposite in the fluorescence intensity rate, glutaraldehyde (25%), pH 4, time 60 s, 350 nm) in Robinson buffer solution.

3.2.3. Effect of time

The Effect of the reaction time on the absorbance spectrum was investigated. Based on Fig. 7, it is apparent that the fluorescence intensity enhanced expeditiously and reached its peak at around 60 s. After 60 s, a relative stability

was spotted in the fluorescence intensity. Thus, 60 s was determined as the optimum reaction time in this experiment [13,16].

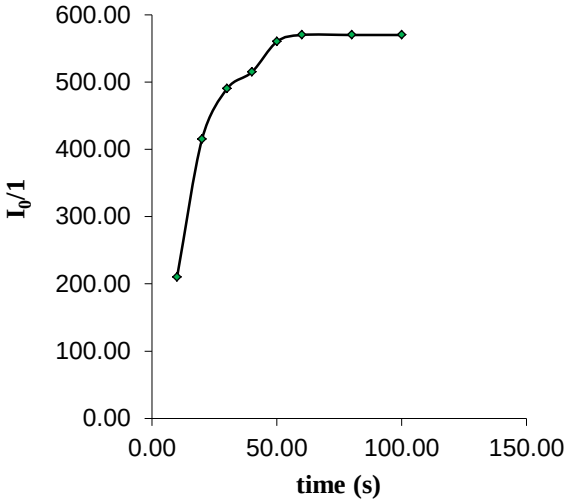

Figure. 7. Effect of time in the fluorescence intensity rate. PbS QDs–Gelatin nanocomposite $^{-۲}$ ۲,۵×۱۰M, glutaraldehyde (25%), pH 4, 350 nm) in Robinson buffer solution.

3.3. Analytical application

Calibration graphs and detection limits: Under the optimum conditions calibration graph was constructed by plotting (I_0/I), values as a function of the toxic abamectin concentration [12,13]. This in section purpose the examined and analytical performance of PbS QDs–Gelatin nanocomposite for determination of toxic abamectin. First, Examined and drawn calibration curve with different concentrations of toxic abamectin from (0.05 to 10.0 µg L^{-1}) using the fluorescence method, which is shown in (Fig. 8). The precision of the method was evaluated by performing 10 replicate measurements of toxic abamectin solutions. The relative standard deviations (RSD) for these determinations were (1.1 %). The limit of detection (LOD) was (0.05 µgL^{-1}), respectively [16,32].

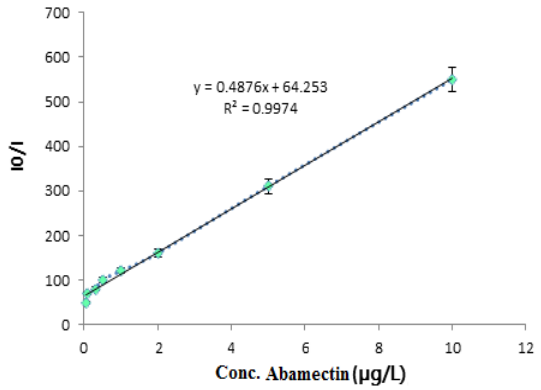

Figure. 8. Calibration curve diagram (0.05–10.0 µg L^{-1} toxic abamectin, ۲,۵×۱۰$^{-۲}$M of PbS QDs–Gelatin nanocomposite, pH 4.0, time 60 s, in Robinson buffer solution.

3.4. Analytical Figures of Merit

The analytical performance of the suggested with fluorescence emission intensity method experiment was evaluated, and the results are summarized in (Table. 1).

Table 1. Investigation of method repeatability at conditions

Parameter	***Optimum Value***
Abamectin (M)	(10.0 µg L^{-1})
PbS QDs–Gelatin nanocomposite (M)	(2.5 × 10^{-2} M)
pH	4.0
Equilibration time (s)	(60 s)
Linear range (LDR)	(0.05 - 10.0 µg L^{-1})
Detection limit (LOD)	(0.05 µg L^{-1})
Relative Standard Deviations (RSD)	(1.1 %)
Advantages	High repeatability, sensitivity, selectivity, wide linear range

3.5. Investigation of competition of toxic abamectin with other ions

The study of was performed in the presence of cations and anions in a solution contained (10 µg L^{-1}) of toxic abamectin in pH 4.0, and Robinson buffer (0.1 M), and 2.5 × 10^{-2} M PbS QDs–Gelatin nanocomposite, time 60 s, and the

tolerance limit was determined as the max concentration of the interfering substance which resulted in an error less than (±5%) for determination of toxic abamectin. The results are shown in (Table. 2) [13,28].

Table 2. Effect of foreign species on the determination of toxic abamectin (n=5).

Foreign species	Tolerance limit (μgL^{-1})
Bentazon, Atrazine, Fenpyroximate	100
Diazinon, Diphenylamine	150
NH_4^+, Mg^{2+}, Na^+, K^+, Ca^{2+}	500
CO_3^{2-}, SO_4^{2-}, Cl^-, I^-	500
Hg^{2+}, Cr^{3+} ,Ag^+, Fe^{2+}, Fe^{3+}	250

3.6. Application in the water samples

The developed method was applied to determine toxic abamectin in water samples. Abamectin from fungicide was used to validate this method by measuring the fluorescence spectrum of QDs solution under optimum condition. The results are shown in (Table. 3). A recovery study was also achieved in order to evaluate the accuracy of the method. It was carried out by spiking toxic abamectin concentrations to the analyzed in real sample [33,34].

Table 3. Detection of toxic abamectin in water samples sample using the proposed method (n = 3).

Samples	Added ($\mu g\ L^{-1}$)	Founded ($\mu g\ L^{-1}$)	RSD %	Recovery %
Fahlyan River	0.00	4.2	1.1	----
warer Fars	10.00	14.1	1.4	99.0
Karon River	0.00	2.8	3.7	----
warerAhvaz	10.00	13.1	2.8	103.0
Karkheh dam	0.00	5.0	3.8	----
water	10.00	15.1	3.0	101.0

4. CONCLUSIONS

PbS quantum dots–gelatin nanocomposite were successfully used as a sensitive and selective fluorescence probe for detection of residue toxic insecticide abamectin for the first time. This sensor is based on the fluorescence quenching effect of toxic abamactin which interacts with functionalized PbS quantum dots–gelatin nanocomposite. Applying provides a sensors based on PbS QDs–Gelatin nanocomposite for the determination of toxic abamectin in water samples. The calibration curve was linear in the range of (0.05–10.0 μgL^{-1}). The standard deviation method (0.05 μgL^{-1}) for toxic abamectin was obtained for the proposed PbS QDs–Gelatin nanocomposite sensor, respectively. The application of the sensor in the natural water sample and its validation with the standard addition method for toxic abamectin detection confirms its authenticity for application in nearly every type of water. PbS quantum dots–gelatin nanocomposites sensor provides several advantages such as simple, mild condition, easy workup, and excellent yield in a short time. Therefore; this method can be provided with excellent selectivity and sensitivity for fast, simple and accurate detection of residue toxic insecticide abamectin in different water samples.

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DISCLOSURE STATEMENT

No potential conflict of interest was reported by the author(s).

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مجله شیمی فیزیک و شیمی نظری
دانشگاه آزاد اسلامی واحد علوم و تحقیقات
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چکیده

در این مطالعه رفتار باقیمانده حشره کش سمی آبامکتین بر روی آب‌های سطحی با حسگرهای شیمیایی فلورسنت به دلیل غیر مخرب بودن، توانایی نشان دادن غلظت‌های تجزیه شده، پاسخ سریع و بالا مورد بررسی قرار گرفت. در این تحقیق، یک حسگر شیمیایی نانو کامپوزیت سولفید سرب با پوشش ژلاتین برای آبامکتین سمی سنتز شد. منحنی کالیبراسیون خطی در محدوده (۰/۰۵تا ۱۰/۰میکروگرم در لیتر) بود. انحراف استاندارد کمتر از (۱/۱ درصد) و محدودیت‌های تشخیص روش (۰/۰۵ میکروگرم در لیتر) در زمان ٦۰ ثانیه، و طول موج ۳۵۰ نانومتر برای پاسخ سطح حسگر سنسور نانو کامپوزیت سولفید سرب با پوشش ژلاتین با (۹۵ درصد)، اطمینان به دست آمده ارزیابی شد. نتایج مشاهده‌شده بازیابی مناسب و حد تشخیص بسیار پایین برای اندازه‌گیری آبامکتین سمی را تأیید کرد. سنسور شیمیایی نانو کامپوزیت سولفید سرب با پوشش ژلاتین به عنوان حسگر عالی در کاربرد عملی آبامکتین سمی مرتبط با مدیریت پسماند در نمونه‌های آب است. روش فلورومتری را می‌توان برای تخمین PHI مناسب استفاده کرد و همچنین می‌تواند از مجوز محصولات حفاظت از گیاه به عنوان اطلاعات تکمیلی پشتیبانی کند.

کلید واژه‌ها: آبامکتین، حشره‌کش، فلوئورسانس، نانو کامپوزیت سولفید سرب با پوشش ژلاتین، اتصالات کوانتومی عرضی

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