iJRASET
International Journal For Research in Applied Science and Engineering Technology



INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 14 **Issue:** XI **Month of publication:** November 2026

DOI: https://doi.org/10.22214/ijraset.2026.85694

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 14 Issue XI Nov 2026- Available at www.ijraset.com

Surface Passivated Carbon Dots as Sensors for monocrotophos-an Organophosphate Pesticide

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Abstract: ***Self-poisoning using pesticides is the most common means of suicide in India. In such cases, the identification of pesticides is of utmost importance in managing the treatment and applying forensic protocols. In the present work fluorescent carbon dots using phenyl alanine as precursor was synthesized and characterized using FT-IR, NMR and TEM. The carbon dots showed enhanced fluorescence in the presence of organophosphate pesticide monocrotophos. Linearity studies showed a detection limit of 0.13 µM. The carbon dot sensor showed good selectivity in the presence of other pesticides and good stability in the acidic pH.***

Keywords: Phenyl alanine, carbon dots, surface passivation, monocrotophos, fluorescence sensor.

I. INTRODUCTION

Self-poisoning using organophosphate (OP) pesticides accounts for more than 60% of the suicides cases in India [1,2]. The major reason is the easy availability of these pesticides in local stores and routine presence in farming households [3]. According to the World Health Organization (WHO), organophosphates (OP) pesticides belong to a class of highly hazardous pesticides [4]. Amongst the OP pesticides, monocrotophos (Figure 1) is extensively used in India, due to its low cost and rapid action [5]. Monocrotophos is a fast-acting cholinesterase inhibitor with systemic effects and is categorized as a class I hazardous substance by the U.S. Environmental Protection Agency [5, 6].

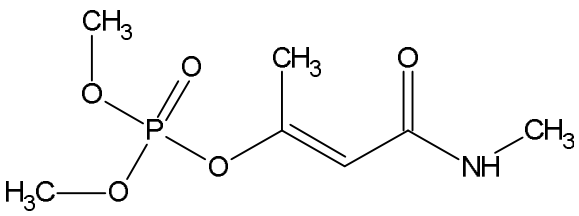


Fig 1 The structure of monocrotophos

In the cases of self-poisoning using pesticides, the accurate identification of the pesticides is important to administer appropriate treatment and ensure legal compliance. As per current forensic protocol, in case of suspected suicidal poisoning, biological and other evidence is initially screened for the presence of the pesticides; followed by advanced instrumental analysis like GC-MS or LC-MS to confirm the pesticide. There are several other methods reported for the sensing of monocrotophos like electrochemical methods [7-10] and optical methods [11-15]. However, these methods are highly sensitive to matrix interferences and use reagents requiring laborious synthesis, specialized instrumentation and labour-intensive workflows.

In the present method, we have reported the synthesis of phenyl alanine based carbon dots (PhA-CDTs) for the selective and sensitive detection of monocrotophos. The carbon dots are easily synthesized by the thermal oxidation method and can be lyophilized and stored as a powder. The detection method requires dispersion of the CDTs in methanol and the visualization of the reaction in a UV chamber.

II. MATERIALS AND METHODS

A. Chemical and reagents

The chemicals and reagents used were purchased from commercial suppliers and used without further purification.

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B. Instruments

The FT-IR spectrum was recorded on Alpha-Bruker Fourier Transform Spectrometer. The proton NMR was recorded on the Bruker 400 Ascen NMR spectrometer (400 MHz) using DMSO-d_6 as the solvent and TMS as reference. The TEM images were acquired on a Thermo Fischer Talos L120c G2. All fluorescence spectra was recorded on a Fluoromax-4-spectrofluorometer (Horiba Japan)

C. Synthesis of the carbon dots(PhA-CDTs)

Hydrophobic photoluminescent CDTs were synthesized by a thermal oxidation process in air using citric acid and L-phenylalanine as precursor molecules in a molar ratio of 1:3. To an aqueous solution of acidified citric acid (0.576gm in 30ml 10% HCl) and aqueous solution of L-phenylalanine (1.48gm in 30ml distilled water) was added slowly in small portions under constant stirring. The resulting solution was evaporated at 100–120 °C to yield a colorless powder. The dried colorless powder was put in a silica crucible and subjected to thermal oxidation in air for 30 min at temperatures of 220 °C.

The end product received was a dark brown coloured viscous liquid which indicates the formation of carbon dots. After cooling it for 30 mins it resulted in formation of dark brown solid product, which was dissolved in acetone and filtered in order to separate undissolved components. The obtained dark brown filtrate was then subjected to rotatory evaporation in order to produce phenylalanine passivated carbon nanodots. The yield received was of light brown colored powder (548 mg).

D. Fluorescence Studies

Methanol was used as the solvent for the fluorescence studies. The stock solution of the synthesized PhA-CDTs prepared in methanol was of 10.84 ppm. The pesticide samples were all 10^{-2} M prepared in methanol and diluted with methanol as per requirement.

III.RESULTS AND DISCUSSION

A. The synthesis and characterization of the PhA-CDTs

The PhA-CDTs were prepared by modifying a method reported earlier [16]. The CDTs synthesized by this method have citric acid as their carbogenic core and phenyl alanine is passivated on the surface of the CDTs as shown in figure 2

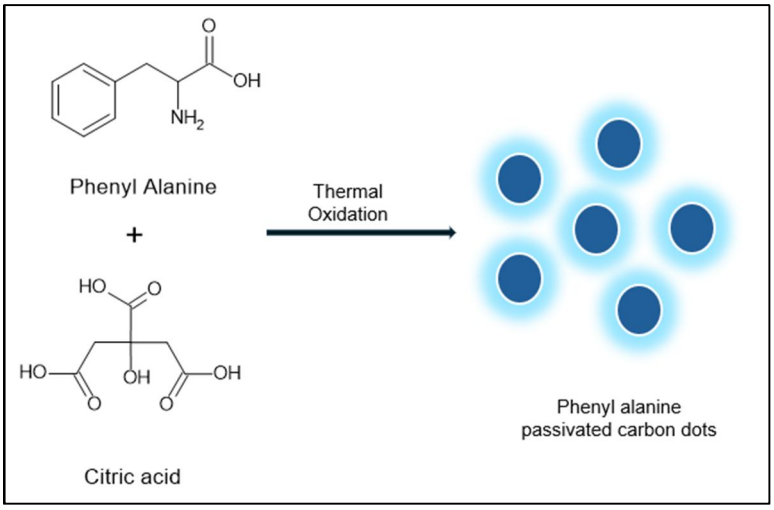


Fig 2. The synthesis of surface passivated carbon dots

The CDTs were characterized by FT-IR, proton NMR and TEM analysis. The TEM image (Figure 3) reveals ultra-small, homogeneously dispersed nanoparticles having an average diameter of 2–5 nm. The particles have a quasi-spherical to spherical "dot-like" morphology.

The FT-IR spectra (figure 4) clearly indicate the functional groups present on the surface of the CDTs. The peak at 3440 cm^{-1} corresponds to the hydroxyl of the group. The merged peaks at 1600 and 1782 cm^{-1} indicated the carbonyl of the carboxylic acid. The proton NMR spectrum (Fig 5) reveals a cluster of peaks centred on ~7.2 ppm characteristic of aromatic protons; the peaks at ~3.3 ppm and ~2.7 ppm are all indicative of the presence of phenyl alanine on the carbon dots

Thus from the FT-IR and NMR spectra the surface passivation of phenyl alanine on the CDTs is confirmed.

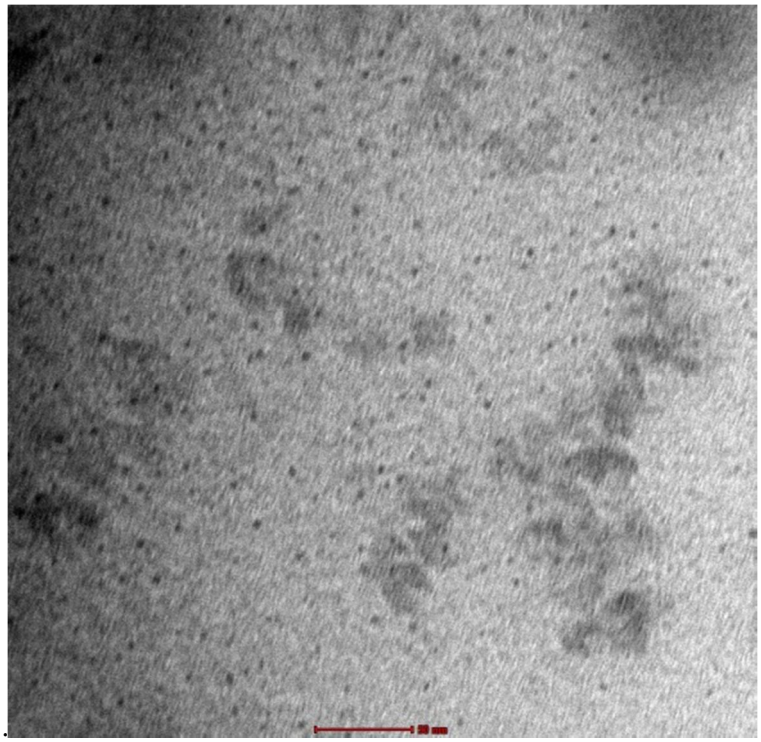


Fig 3 TEM images of PhA-CDTs

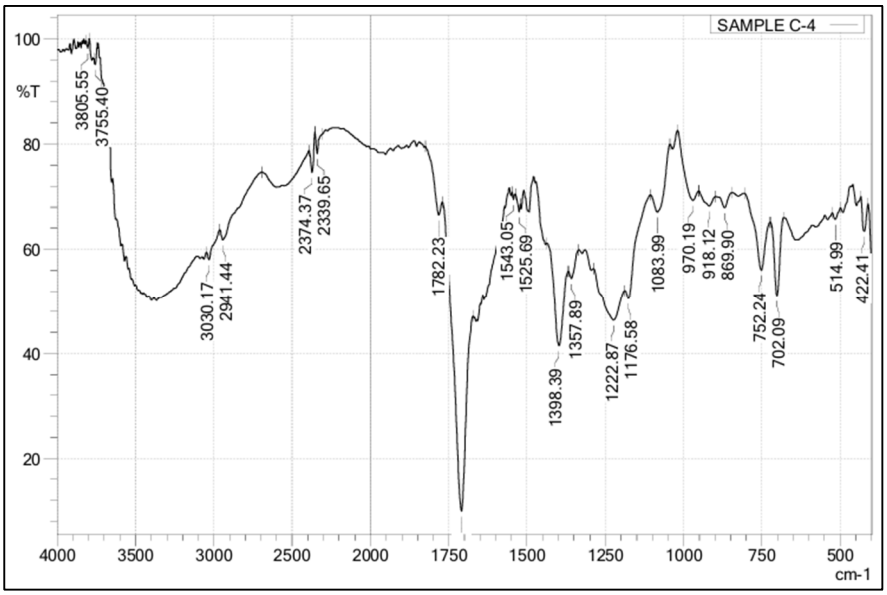


Fig 4 FT-IR spectra of the PhA-CDTs

B. Selective sensing of Monocrotophos

CDTS showed a faint fluorescence at 428 nm. On interaction with various pesticides, it was observed that the fluorescence was significantly enhanced by monocrotophos. The percentage enhancement in the fluorescence intensity for all pesticides on interaction with PhA-CDTs is as shown in Fig 6

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
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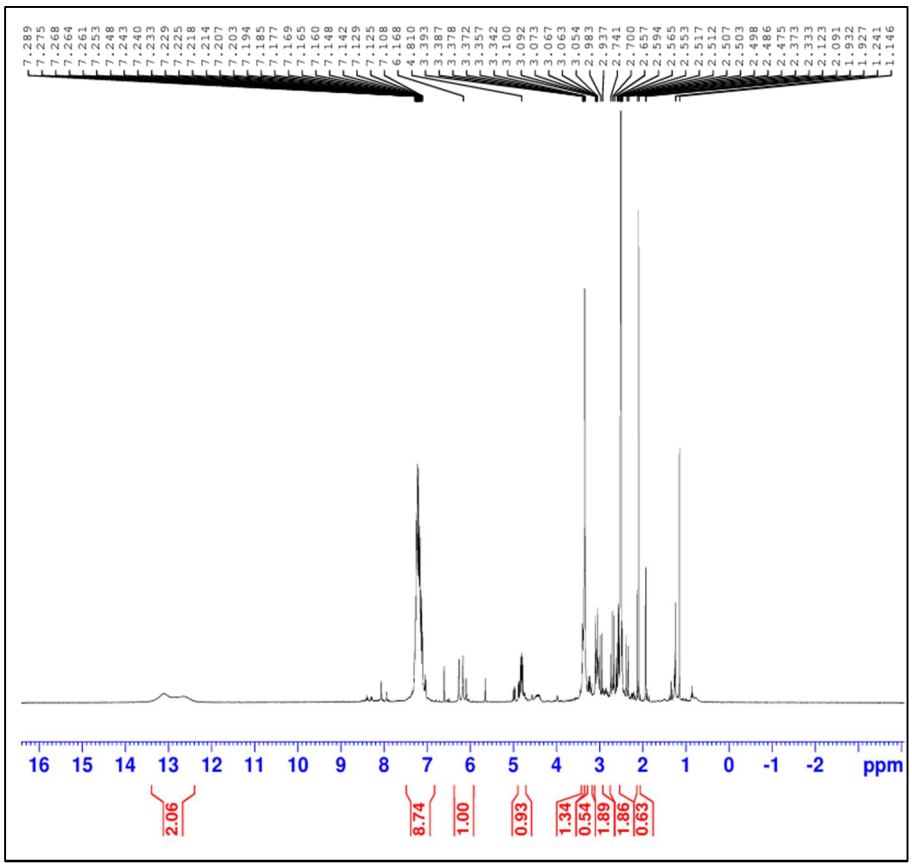


Fig 5 ^{1}H-NMR spectra of PhA-CDTs

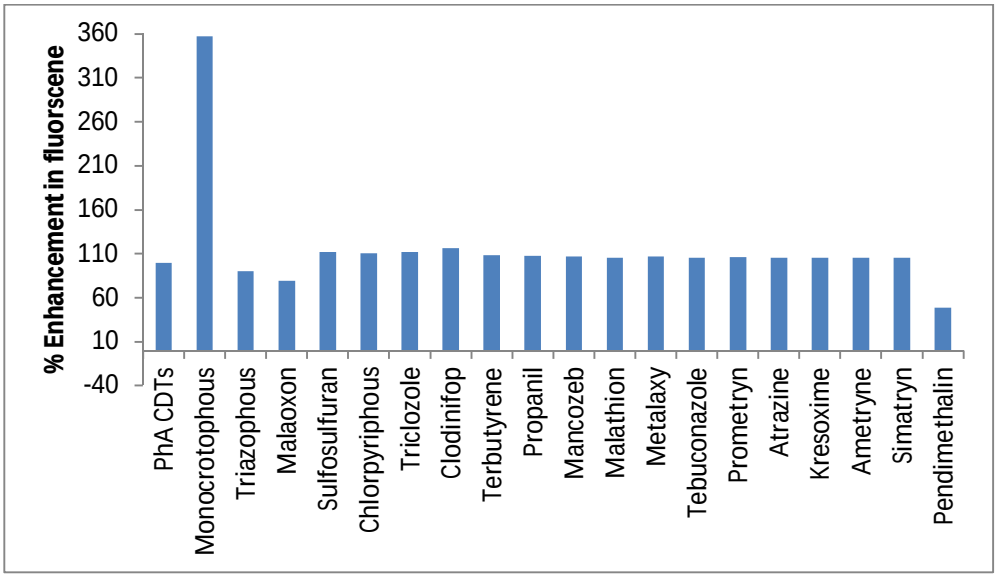


Fig 6 Percentage enhancement in fluorescence of PhA-CDTs on interaction with various pesticides

The sensitivity of the CDTs sensor was studied by measuring the increase in fluorescence with increasing concentration of monocrotophos (Fig 7).

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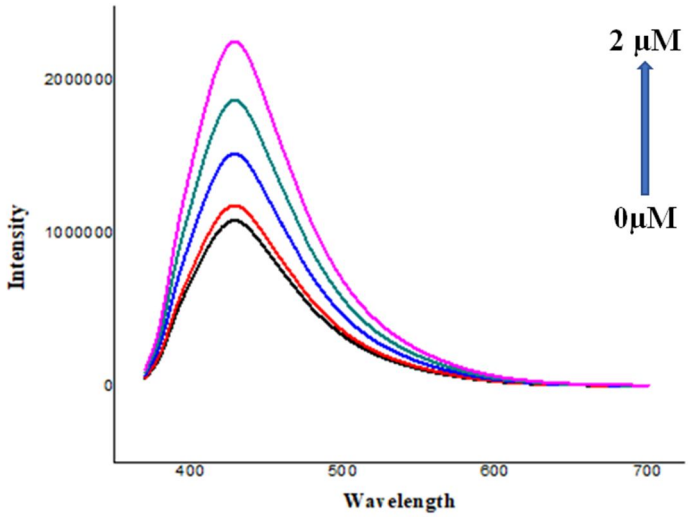


Fig 7 The effect of increasing monocrotophos concentrations on the emissions intensity of PhA-CDTs

As shown in the Fig 8, the plot of F-F_0/F vs Q is linear in the concentration range of 0-20 µM. The limit of detection was calculated to be 0.13µM. To understand the binding of monocrotophos with the CDTs, binding constant K_b and the binding stoichiometry n were evaluated using the equation shown below [16-18]:

$$log\frac{F_0 - F}{F} = logk_b - nlog[Q]$$

Here, F_0 and F are the emission intensities of the CDTs in the absence and presence of monocrotophos respectively, [Q] is the concentration of monocrotophos, K_b is the binding constant and n is the stoichiometry of the binding.

Table I
Different Parameter For The Interaction Of Monocrotophos with PHA-CDTS

Parameter	**Value**
K_b ($L.mol^{-1}$)	1.406 x 10^6
n	1.1
Limit of detection (µM)	0.13

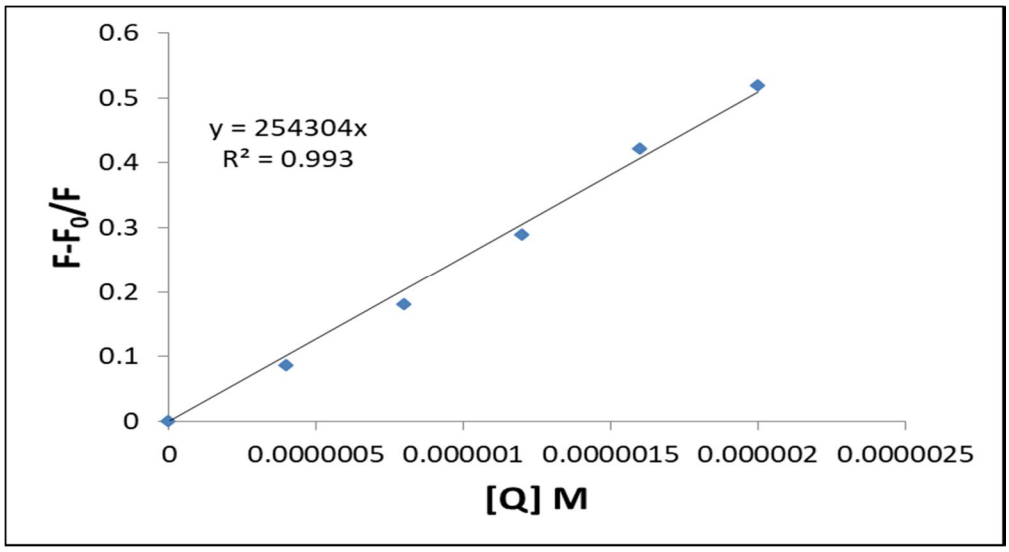


Fig 8 Plot of F-F_0/F vs [Q] for the interaction of monocrotophos with the PhA-CDTs

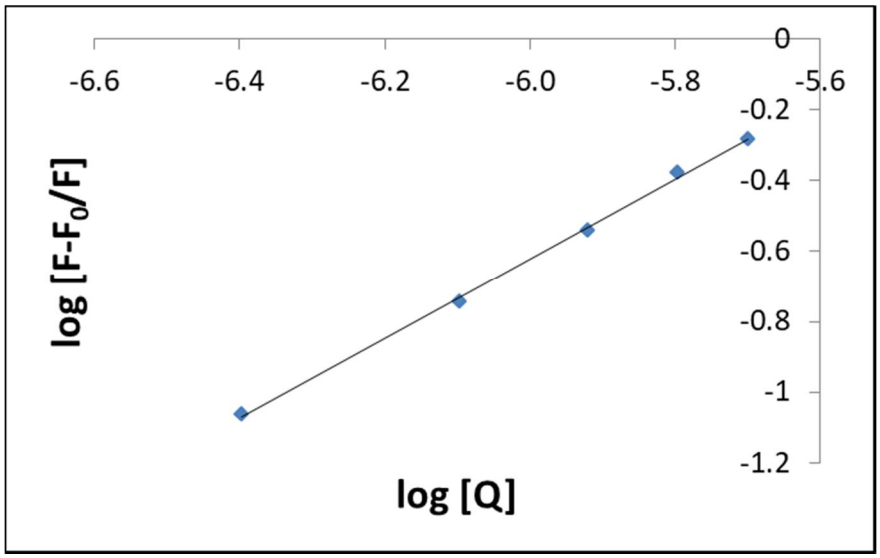


Fig 9 Plot of log F-F0/F vs log [Q] for the interaction of monocrotophos with the PhA-CDTs

C. Stability of the PhA-CDTs

The CDTs were subjected to pH changes from 3 to 10 and the emission intensity measured at each pH (Fig 10). As shown in the Figure 10, the emission intensity changes appreciably above pH 7. In the alkaline pH, phenylalanine may undergo changes in its functional groups causing the decrease in the emission intensity [19]. Hence, we may conclude that sensing studies needs to be carried out below neutral pH. Alkaline matrices may have to be neutralized before sensing studies are done.

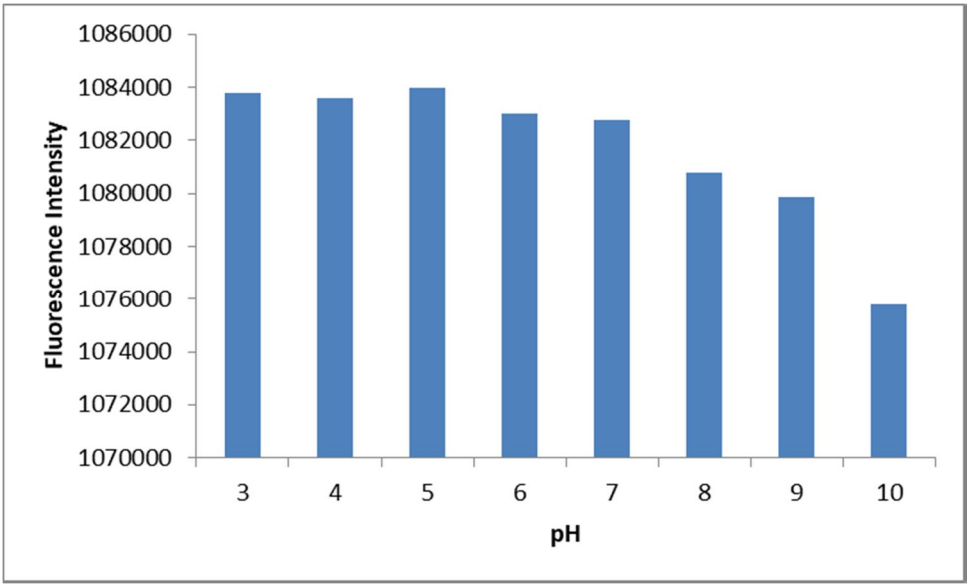


Fig 10 Emission intensities of PhA-CDTs at various Ph

IV. CONCLUSIONS

In summary, phenyl alanine based carbon dots synthesized by a thermal oxidation method show weak fluorescence at 428 m; the emission intensity is enhanced when the CDTs interact with monocrotophos. The CDTs show excellent selectivity towards monocrotophos and are useful in the pH range of 3-7 for sensing purposes. The limit of detection for the reported method is 0.13 µM which is significantly higher compared to some recently reported methods (Table 2). But when the sensor materials are compared, the CDTs in the present work are low cost and synthesized by a simple methodology. They can be lyophilized and stored for a long time. Moreover, the sensing method is also simple requiring no specialized equipments and can be easily performed on-site for forensic and environmental samples.

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TABLE III
DIFFERENT PARAMETER FOR THE INTERACTION OF MONOCROTOPHOS WITH PHA-CDTS

Sr. No.	Sensor material	Technique	Limit of detection for monocrotophos	References
1	Indoxyl acetate (IDA) immobilized on silico alcogel	Colorimetric sensor	0.0036 µM	12
	5,5′-Dithiobis (2-nitrobenzoic acid) (DTNB) immobilized on silico alcogel		0.0022 µM	
	α-Naphthyl acetate (α-NAc) immobilized on silico alcogel		0.0019 µM	
2	Bisnaphthylated-azacalix[4]arene	Fluorescence sensor	5.5 µM	13
3	Fe(III) Salophen complex	RLS and Fluorescence sensor	0.03 µM	14
	Eu(III) Salophen complex		0.4 µM	
4	NH_2-UiO-66@cur	Fluorescence “turn-on”	0.0018 µM	20
5	Graphene nanoplatelet-based SELEX	Aptamer-based electrochemical sensor	0.0026 µM	21
	Graphene oxide-based SELEX		0.0035 µM	
6	Molecularly imprinted polymer (MIP) on the surface of a screen-printed carbon electrode (SPCE) which consists of polyvinyl alcohol (PVA), glutaraldehyde, citric acid and Fe_3O_4	Electrochemical sensor	0.045 µM	22
7	Zirconium-terephthalic acid combined metal organic framework	Electrochemical sensor	0.0038 µM	23
8	Acetylcholinesterase on chitosan and CNTs modified carbon paste electrode	Electrochemical sensor	0.0003 µM	24
9	Anti-monocrotophos antibodies conjugated with graphene oxide (GO layered onto a fluorine-doped tin oxide (FTO) electrode	Electrochemical sensor	2.19 µM	25
10	Phenyl Alanine Carbon dots	Fluorescence sensor	0.13 µM	Present Work

V. ACKNOWLEDGMENT

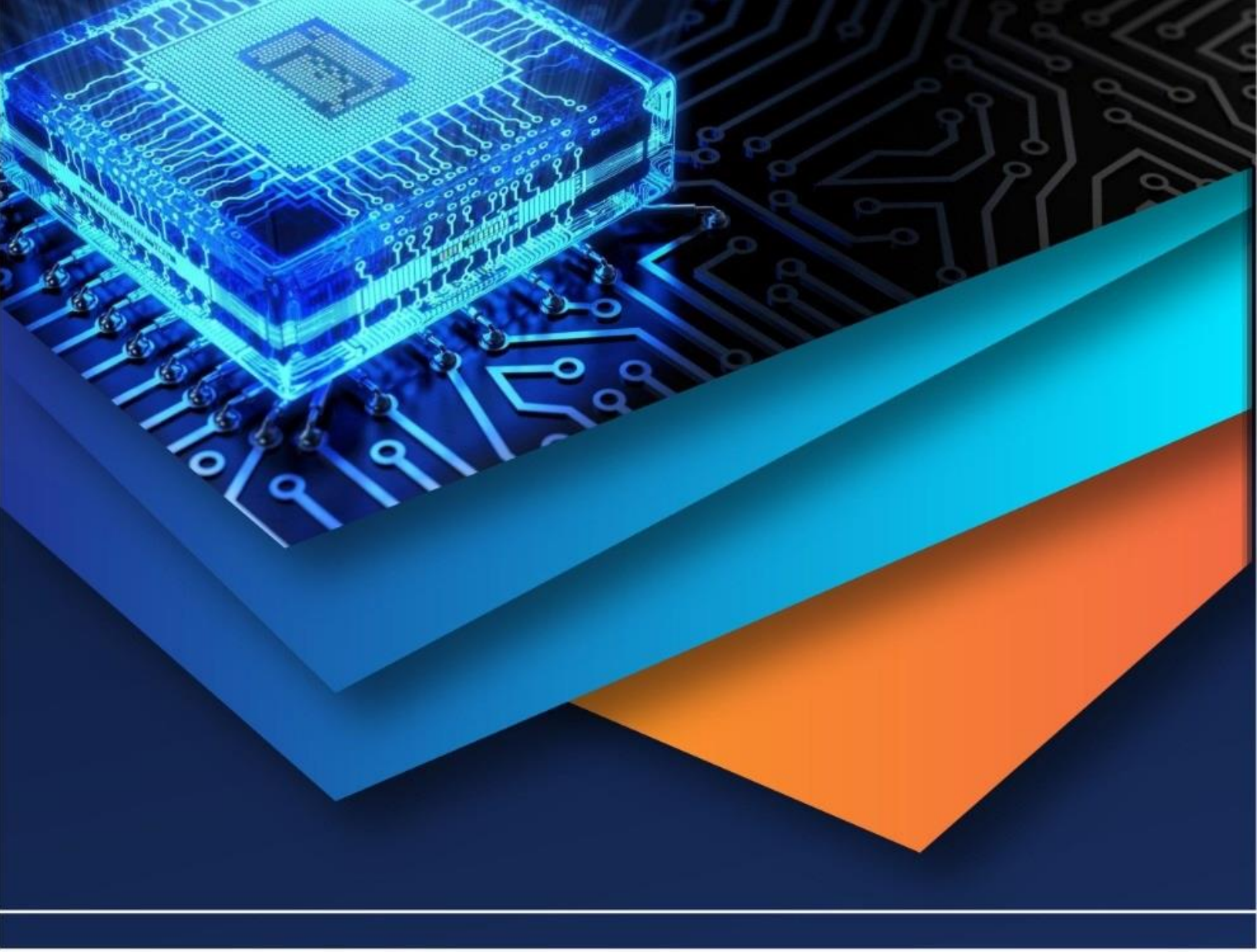
The authors are grateful to the Department of Biochemistry and Forensic Sciences for providing all facilities including chemicals and instruments. Ms. Devanshi Bhatt is grateful for the funding provided from SHODH- Scheme of Developing High Quality Research Scholarship (Reference Id: 202001380098).

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