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Forensic Detection of 1,3- dinitrobenzene with Dipyrene Oxacalixarene(DPOC)

Himali Upadhyay[1], Uma Harikrishnan[2], Devanshi Bhatt[3], Namrata Dhadnekar[4], Kapil Kumar[5]
[1,3,4,5]*Department of Biochemistry and Forensic Science, Gujarat University, Navrangpura, Ahmedabad- 380009*
[2]*Department of Chemistry, St. Xavier's Collage, Navrangpura, Ahmedabad- 380009*

Abstract: Crime related to explosive and explosive related components are increasing rapidly. To combat the devastating effects of global terrorism, the detection of explosives and explosive-related materials is presently one of the most pressing global concerns. In recent years, significant progress has been made in the development of fluorescence-based chemical sensors for the recognition of explosives in the solid, solution, and vapor phases, with improved sensitivity, selectivity, and response speed. Quick, sensitive and selective detection of explosive is very much important in nowadays. In this paper, DPOC molecule is synthesized and characterize for detection of 1,3- dinitrobenzene. Detection limit is 7 µM. This method can be a good alternative for on-site detection of 1,3- dinitrobenzene at crime scene.
Keywords: Explosives, 1,3- dinitrobenzene, Forensic Science, Calixarene, Crime Scene.

I. INTRODUCTION

In the past two decades, nitroaromatic compounds (NAC) have attracted considerable attention due to their severe toxicity. The production, storage, and testing of explosives, as well as their use in terrorist and military activities, pose a serious threat to global public safety [1][2] As an important pollutant, NAC causes health issues in both animals and humans, such as anemia, aberrant liver function, cataract formation, and skin irritation [3]. Even after degradation, the by-products of common explosives continue to be carcinogenic and toxic [4]. Consequently, public security specialists and environmental scientists are becoming increasingly interested in the identification and quantification of NAC. 1,3-Dinitrobenzene (DNB) is a nitroaromatic explosive that is extensively employed in military munitions, civilian mining, and terroristic activities. DNB is also an important organic raw material and has been extensively adopted in the chemical synthesis industries for the production of numerous intermediates such as aniline, quinoline, azobenzene, and trinitrotoluene. These intermediate products are then utilized in the production of explosives, rubbers, pesticides, agricultural compounds, and solvents for coating materials and dyes. Due to its extensive applications, DNB is inevitably exposed to humans and the environment on a regular basis. In addition, DNB is a member of the carcinogenic and persistent organic pollutant class, resulting in health concerns for a variety of organisms due to its toxicity and persistence in ecosystems. DNB is commonly used to induce experimental brain stem lesions and testicular damage in rodents[5]. Occupational exposure to DNB in humans can induce anaemia, nausea, neurological symptoms, and liver damage. Therefore, the sensitive, selective, and cost-effective detection of DNB is essential for modern homeland security, food safety, and environmental protection. From fluorescence [6] and luminescence [7] to FTIR spectroelectrochemistry [8]. Today, numerous sensors and detection methods for nitrobenzene, including DNB, have been developed. Despite significant advancements in the sensing of DNB, a portable and reliable method for sensitive detection in a field environment still encounters significant obstacles, including interference from common household and personal care products. In this paper, we have developed a new method for selective and sensitive detection of 1,3- DNB with calixarene. Calixarene are essential structural components in supramolecular chemistry. The lower rims of the upper and lower extremities are functionalized, providing excellent binding sites for the encapsulation of guests and the assembly of molecules [9].

II. MATERIALS AND METHOD

For the synthesis of receptor Sigma-Aldrich was used to get all of the necessary compounds, such as 1,5-difluoro-2,4-dinitrobenzene, Phloroglucinol, and NACs. All of the Finar Chemicals (AR grade) solvents used in the synthesis and analysis were purchased and used without further purification.

A. Synthesis of Receptor (DPOC)

For the synthesis process, at room temperature, the reaction is conducted by Potassium carbonate and oxacalixarene in dry acetone solution [10]. After that add 3 bromo methyl pyrene and mix it vigorously for 24 h. In a vacuum, the solvent is exhausted.

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The obtained crude is subjected to silica gel column chromatography with EtOA-C: Hexane (3:7) as the eluent. DPOC was produced by separating, drying, and recrystallizing the fractions in alcohol.

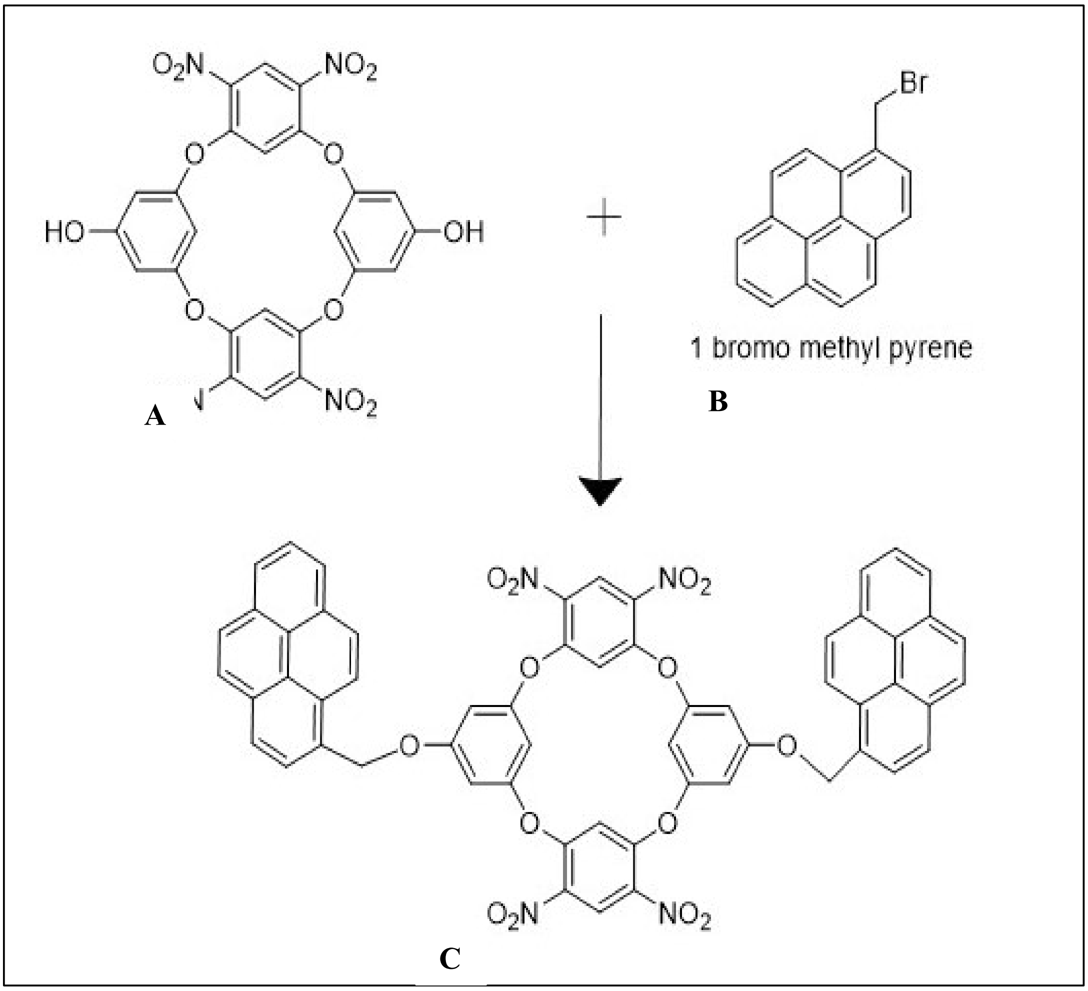

Figure 1 Synthesis process of DPOC, in which A represents Oxacalixarene, B represents 1 bromo methyl pyrene and C represents formed receptor DPOC

Here ^{1}H NMR result is shown as a characterization purpose, which proves the complexation of the molecule. Results of analysis: (500 MHz, DMSO d6): δ= 10.22 (s, 4H); 7.83 (d, 1H), 7.76 (d, 1H), 7.91 (d, J=8.1 Hz, 4H); 7.33 (m, 8H); 5.88 (2H, s).

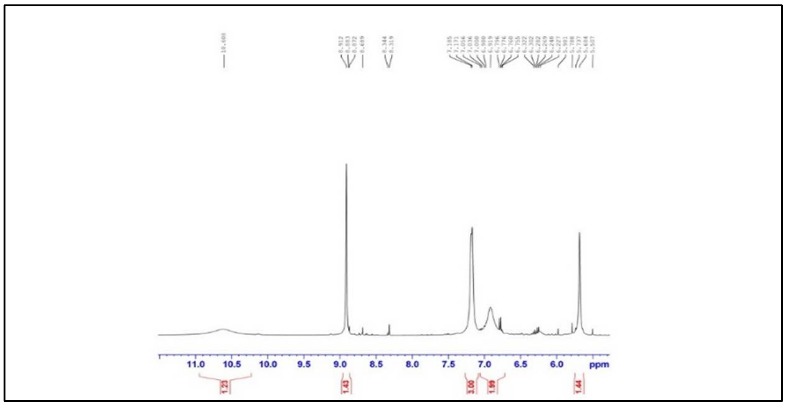

Figure 2 ^{1}H NMR of formed molecule

B. Application of Receptor

With the aid of spectrofluorometric measurements, the NAC-sensing capability of the DPOC probe was investigated. The stock solution of the receptor DPOC was initially prepared with Acetonitrile as the solvent. In order to conduct spectrofluorometric experiments, the stock solution was further diluted to 100 M. Similarly, stock solutions of explosives including 1,3- DNB, 2,3-DNT, 2,4- DNT, 2,6- DNT, 4-NP, and 4- NT. Only 1,3- DNB reacts with DPOC and shows change in fluorescence intensity. Lower detection limit is up to 7 µM. Here is the mass analysis data of formed complex (Fig.4).

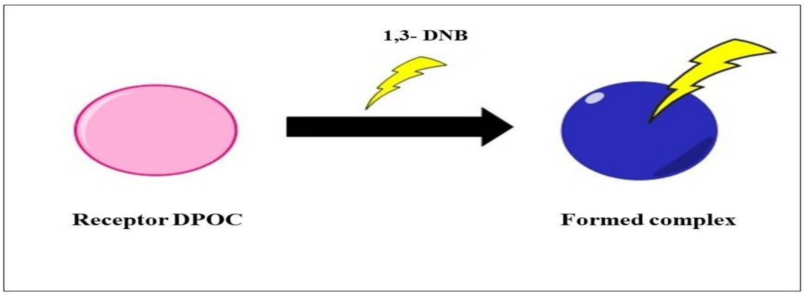

Figure 3 Graphical deception of reaction procedure between receptor and 1,3- DNB

C. Results of Reaction Procedure

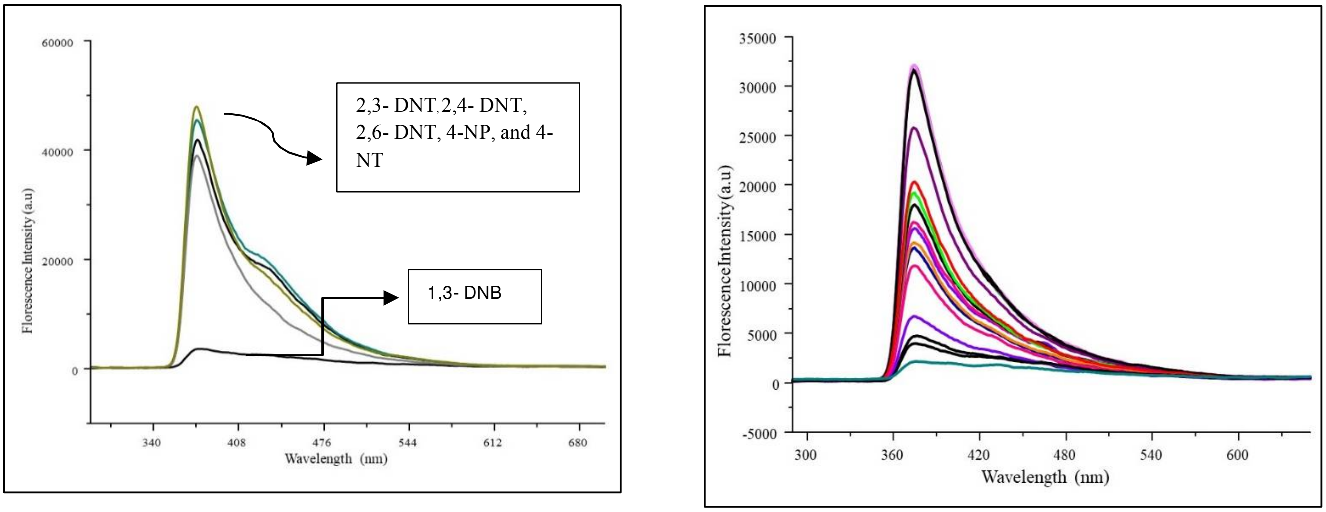

Figure 4 Selectivity of DPOC towards 1,3- DNB and linear concentration graph of DPOC and 1,3 DNB

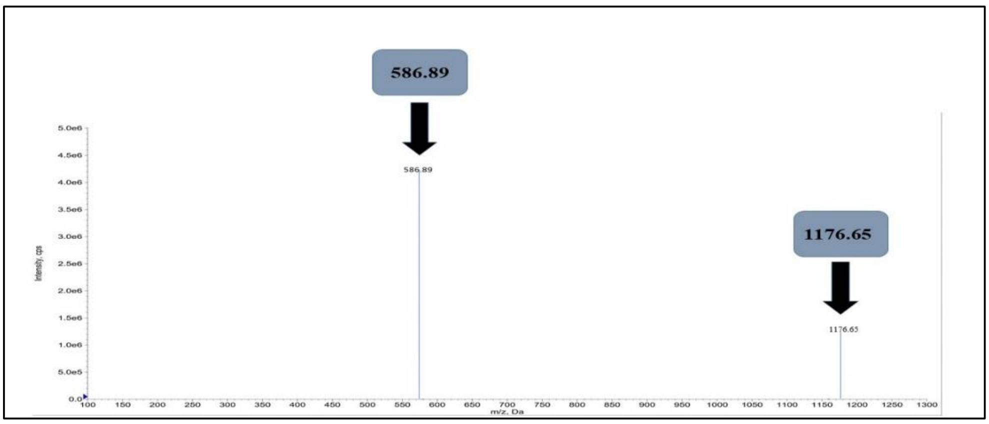

Figure 5 Mass analysis of DPOC and 1,3- DNB

III. CONCLUSION

As a component of improvised explosive devices (IEDs) or as a precursor in the manufacture of illegal explosives, 1,3-dinitrobenzene may be utilized. In order to identify the presence of this compound at crime sites, trace its origin, and collect evidence for legal proceedings, its detection is crucial in forensic investigations. Sensitive and selective detection techniques aid law enforcement agencies in their fight against terrorism, criminal activity, and illegal explosives production. Calixarenre can be a good alternative for sensor development for detection of 1,3 – dinitrobenzene because of their good selectivity and sensitivity.

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