

Research Article

Employing a Carbon-Based Nanocomposite as a Diffusive Solid-Phase Extraction Adsorbent for Methamphetamine for Therapeutic Purposes

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Received 27 August 2022; Revised 5 October 2022; Accepted 10 October 2022; Published 14 April 2023

Academic Editor: Debabrata Barik

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Due to the obvious minimal doses of drugs in biological matrices as well as the societal difficulties caused by methamphetamine usage, methamphetamine identification is critical in clinical and forensic laboratories. Because of their simple and inexpensive production procedure, as well as their excellent selectivity and sensitivity, polymeric carbon-based nanocomposites are strong contenders for the diffusive solid-phase extraction approach. The diffusive solid-phase extraction adsorbent nanographene oxide polypyrrole composite was produced and used to recover methamphetamine from a complicated urine substrate. The generated NGPPC was fully characterized, and the significant extracting parameters have been explored using the one-parameter-at-a-time strategy. NGOPC is being used to extract methamphetamine using a urine medium with high efficiency. The NGPPC synthesizing procedure was easy, and the extraction method will demonstrate good repeatability. Moreover, the practical and efficient synthesis process stimulates the use of carbon-based compounds in various extraction procedures. As for detecting and quantifying equipment, HPLC monitors are being used. 300 mL methanol, 7 min extracting and desorption duration, 5000 mixing frequency, urinary pH value of 20, 40 mg adsorption, and 5 mL amount of urine were the optimal extraction variables. Following tracing the calibration graph, the method's linear ranges were determined to be 40-600 ng/mL. The detection limits (LOD) and quantitation limits (LOQ), correspondingly, were 10 and 35.80 ng/mL. The proposed methodology seemed to have a detection range of 9 ng/mL. The suggested approach's applicability in numerous characterization and medical facilities was proven by the examination of addicted subjects using the proposed technique. For successful extraction of methamphetamine using biological urine samples, the carbon-based adsorbent was being used as diffusive solid-phase extraction adsorption.

1. Introduction

A nanocomposite is comprised of two or perhaps more various materials with different physicochemical characteristics, at least one of which is a nanomaterial. Nanocomposite components are formed to have characteristics that far outweigh, and in some cases vastly outweigh, the capacity of its constituent elements. Components (referred to as the reinforcement phase) were embedded in some other materials in order to create nanocomposites. Nanomaterials could exist in one or both stages [1]. The matrices are frequently stretchy or rougher, while the reinforcement chemicals are fairly strong and have a lower density. If the mixtures are effectively designed and manufactured, they combine the strength of the reinforcement with the hardness of the substrate to provide a unique combination of desirable properties not available in any separate traditional materials. The most difficult aspect of manufacturing nanocomposites is creating a uniform diffusion of nanoparticles [2]. The efficiency of the diffusion must have an impact on the interactions among the stages, which could influence the nanocomposite's properties of the resulting. That becomes feasible to tailor distinctive features (such as biomechanical, electrically, thermodynamic, magnetism, or indeed acoustical) by integrating differential materials, architectures, and concentrations in nanocomposites, enabling the nanocomposite substance ideal for diverse applications. As a result, nanocomposites have spawned the rapidly expanding area of multifunctional materials. Nanoparticle fillings contain a limited number of atoms each particle including, as a result, might also have distinct characteristics and significant connections with the matrix over larger materials. It is characterized by a large nanoparticle-matrix interfacial area, as well as molecular basis interconnections among nanoparticles and matrices, which were thought to make a significant contribution to affecting the physical and mechanical features of nanocomposites [3].

Nanocomposites could be made from a variety of components, including nanostructured materials, biomaterials, and conducting polymers [4]. For example, using genetic engineering, the researchers blended two separate organic elements with distinct and crucial features into a single composite structure. They created a new biomimetic nanocomposite by integrating the properties of silken and biosilica through the design, manufacturing, and characterization of a novel group of chimera proteins. Polymer nanocomposites are two-phase mechanisms made up of polymers and reinforcing fillers having a large surface area [5]. The improvements in mechanical characteristics are with very low contents loadings. Nanocomposites are also possible with standard polymeric manufacturing, eliminating the expensive layout necessary for traditional fiber-reinforced composite manufacture. Nanocomposites, with the exception of enhanced elastomers, really have not met expectations [6]. While assertions of a tenfold increase in rigidity exist, these assertions are contradicted by experiments that demonstrate little or no change. At this time, we are only interested in the effect of nanoscale fillers on composites' modulus. The essential possessions of the matrix and filler, as well as inter-

faces among the two, have a role in modulus increase. Poor diffusion, poor interfacial deformation, technique defects, poor alignments, reduced load transmission to the interiors of filling strands, and the fractal structure of filler groups have all been blamed for nanocomposites poor performance. Figure 1 depicts the illustration of the nanocomposite.

Industrial and academic sectors have paid close attention to organic/inorganic nanocomposites. To blend the characteristics of diverse materials and obtain regulated characteristics and prospective applicability, many methodologies were used to create and manufacture organic/inorganic nanocomposites. Due to the obvious possible advantages in chemical sensing, catalytic, optics, and electrical equipment, much effort has been devoted to including or decorating metallic nanoparticles in the matrices or on the surfaces of polymer electrolytes. Polypyrrole (PPy), a conductive polymer with strong environmental resilience, high conductivity, and biocompatibility, is of specific importance [7]. By utilizing various nanostructures, such as nanowire, nanotube, and nanoparticles, PPy was being employed as a matrix to integrate or scatter many metal nanoparticles used in electrocatalysis and sensing. A conjugated polymer with unique electrical characteristics, such as conductivity, is PPy. Additionally, due to their favourable chemical and physical properties, PPy has also developed into one of the most researched materials for biological applications. The aggregating of inorganic nanoparticles, on the other hand, will result in a loss of surface area as well as a reduction in predicted attributes as a result of decreasing overall surface tension. Numerous synthesized approaches have developed in recent years to address this problem. Super capacitor electronic interfaces made of graphene/PPy nanofiber combinations were used to improve its faradaic response, resulting in increased resistance. PPy nanotubes, unlike PPy nanofibers, have an interior cavity that is several to thousands of nanometers in diameter, allowing electrolyte transportation not just to the internal sections of the PPy nanotubes but also with the outer surface. PPy nanotubes have a great potentiality for ionic conduction and are accessible, which means they have a lot of capacitance. At ambient temperature, PPy may be readily produced in large quantities using a variety of liquids. It may be made with a variety of porosities and has a high surface area that could be precisely controlled by adding activating chemicals, rendering it more appropriate for biological applications. Corrosion protection, fuel cells, microsurgical instruments, biosensors, brain tissue engineering, and drug delivery systems are just a few of the applications for PPy today.

Throughout the last twenty years, the nanoscience concept has grown into a wide variability of effect types, and the concentration of nanotechnology/nanomaterial is circumfluence in several promising grounds, such as sensing, biomedical, and numerous helpful implementations. The capability to synthesize nanomaterials from varied structural materials, along with turning the specimens into sophisticated nanoarchitectures, has been accelerated research in related domains [8]. Due to this, owing to its large conduction capabilities, the monolayer of two-dimensional graphene substantial having honeycomb lattice construction,

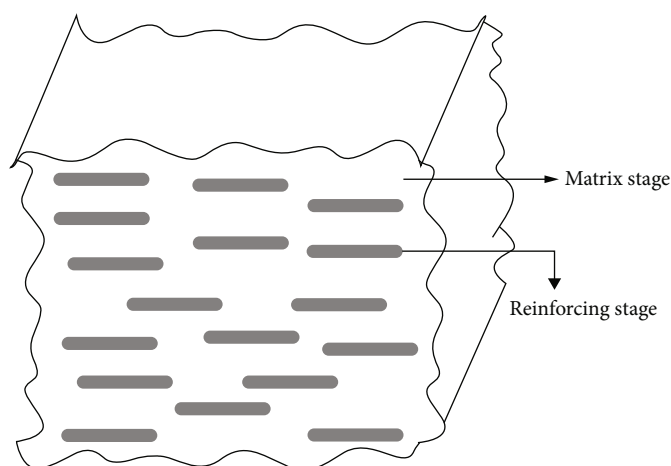


FIGURE 1: Nanocomposite illustration.

that includes an intercarbon binding affinity of roughly 0.142 nm, is widely used in biomedical and nanomedicine purposes. Biological properties which provide for cellular proliferation as well as development, such as the delivery of drugs and chemotherapeutic therapy, might benefit greatly from graphene-based nanomaterials. Furthermore, the materials could be utilized to engage bioactive molecules (e.g., DNA, membranes, or proteins). Graphene/chitosan films have previously been investigated as an implant material in synthetic biology using the solutions fabrication technique [9]. Graphene oxide (GO) that has not been properly functionalized has been discovered to be a hazardous substance. Functionalized nanographene as well as its composite materials is having increasingly garnered attention in biological applications owing to its special and improved physico-chemical characteristics. GO and reduced graphene oxide (rGO), on the other hand, could have been functionalized with biocompatible polymers including PEGylated (PEG), which can be achieved by both covalently and noncovalent techniques to improve physiological environment stability. A sensitivity platform made of graphene is frequently utilized for detecting biological systems in the ability to identify the transition mechanism in addition to the power current of the human cell membrane. The profusion of functionalized nanoscale rGO-based bio-conjugated nanocomposites has already been widely used as drug or gene delivery methods owing to its improved porous structure (single sheet organized with carbon atoms) [10]. Furthermore, due to its strong near-IR (NIR) absorption, its associated compounds of functionalized graphene exhibited remarkable tumor elimination therapeutic benefits. Graphene oxide (GO) is made up of numerous carboxylic, carbonyl, and epoxide structural features and has a higher specific surface area. It was a great biochemical, thermal, and mechanical stability. Furthermore, conductive polymers (CPs), such as polypyrrole, have highly reversible electrochemical activity as well as unique plastic-metal characteristics. Because of their stability and multifunctionality, CPs have gotten a lot of attention in recent decades. Combining different materials can be a good way to make new activated carbons that have the benefits of many of the constituents [11].

Methamphetamine is an extremely addictive amphetamine which generates the consequences. Crystal methamphetamine is a methamphetamine which looks like pieces of glass or shining, bluish-white stones. This has a similar chemical structure to amphetamine, a medication was using to treat ADHD and drowsiness, an insomnia [12]. The United Nations Office on Drugs and Crime reported that 290 tonnes of methamphetamine were produced in 2005, which is equal to 2.9 billion 100 mg doses of antibiotics. Methamphetamine is the two most frequently used illicit substances in the world, with a global prevalence of 0.4 percent. The medicine is most commonly used in Asia, Oceania, and North America. Adult prevalence rates in the Philippines are 14 percent, 3.2 percent in Australia, and 0.8 percent in the United States. Methamphetamine, often known as MA, is a central nervous system stimulant that causes intoxication by enhancing the activity of dopamine and norepinephrine pathways in the brain [13]. To a much lesser extent, it is also used as a treatment for attention deficit hyperactivity disorder and obesity, but recreational usage is by far the more popular application of this substance. The benefits of MA, such as attentiveness, euphoria, and a sensation of well-being, remain significantly longer than those of cocaine, and the drug is processed by the body at a much slower pace. MA is a compound of amphetamine, which had been initially produced in 1887 by a German scientist and researched thoroughly in the early 1930s. Amphetamine is a sympathomimetic medication that activates the compassionate division of the autonomous nervous system, comparable to ephedrine [14].

Muscle breakdown, neurosis, delusions, and seizures are all frequent side consequences through methamphetamine use. Suicide, road accidents, and violence are all important social issues brought on by large doses. In medical and forensic laboratories, organic fluids (urine and plasma) are common samples. Urine is the most useful of these fluids since it is readily available, is intrusive, and can be prepared in huge quantities. Methamphetamine is eliminated in urine at a rate of 37–54 percent in a pH range of 6–8. Because of this, it is quite likely that methamphetamine will be found in the individual's urine [15]. Because of the obvious

complexity and small doses of the analyte in the urine medium, conventional urine analysis is not applicable in the majority of situations; as a result, the development of a technique that combines high sensitivity and selectivity is essential. However, a standard urine test can identify methamphetamine in the system for anywhere from three to five days after the last dose was taken. Because of the composite matrix and low dosages of analytes in varied biochemical media, chemical analysis requires the construction of innovative sample processing techniques. Solid-phase extraction-based (SPEs) approaches being used in analysis methods include solid phase microextraction (SPME), dispersive solid phase extraction (DSPE), magnetic solid phase extraction (MSPE), and microsolid-phase extraction (M-SPE). It has been demonstrated that utilizing these approaches results in high analyte recoveries while using a little number of desorption liquids and achieving optimal preconcentration parameters. Adsorbents were substances that are used in SPE to extract or remove medicines or contaminants from aquatic or biological composites [16]. Solid-phase extraction is a class of alternate extraction procedures (SPE). SPE is a broad field with much applicability that has been the focus of countless articles and research. In most cases, a watery capacity to identify through an immobilized stage is during the distillation process before being extracted with suitable organic solvents. For extremely polar analytes, nevertheless, reduced performances due to poor retention, resulting in low breakthrough volumes, can be seen. Nonetheless, changing the kind of sorbent is among the SPE techniques for overcoming breakthrough quantity [17]. The construction consisting of a polypropylene cartridge with an inserted adsorption stage is the one that is utilized the most frequently in SPE. A high surface area melt-blown polypropylene media is used in the construction of the PP Cartridge. This allows for a minimal initial pressure drop, a high dirt holding capacity, and high-efficiency performance. The substances to be separated are divided into two phases: a solid phase (bed sorbent) and a liquid-liquid phase in SPE (sample). The solid phase should have a higher specificity for such compounds than the chromatographic matrix. Column preparations, specimen load, columns post-wash, and specimen adsorption are the 4 phases of SPE in particular. The stationary phase is conditioned using the abovementioned prewash process. In particular, the post-wash is used to remove unwanted materials. Following washing off the interference chemicals, the targeted analytes are maintained on the suitable bed sorbent. After that, the appropriate elution solvents were utilized to retrieve the data [18].

The differentiation strategy of analytes between the granular packaging and the liquids moving phases is the basis for SPE extraction. A miniaturized extraction (micro-SPE) has many benefits, including the ability to connect directly to high-performance liquid chromatography (HPLC), gas chromatography (GC), or capillary electrophoresis (CE), lower operating costs and time, and the ability to be partially or fully automated, allowing for higher repeatability and hyphenation. SPE's fundamentals entail the separation of substances into two components. Because they should utilize

a higher similarity again for the solid stage than the model matrix, the analytes that need to be extracted are separated between the solids and the liquid phase in the SPE procedure (retention or adsorption step). Solid phase extraction is a technique that takes use of the difference in affinity between an analyte and interferences that are present in a liquid matrix (sorbent). Substances that have remained on the solid matrix could be eluted using a solution that has a higher affinity for such analytes at a later phase (elution or desorption step). Intermolecular interactions among the solution, the adsorption sites on the adsorbent surface, and the dispersion medium or matrix were responsible for the various processes of detention or extraction. The processes underlying in columns column chromatography are about the same. The term HPLC stands for high-performance liquid chromatography. "Chromatography" is a process that separates, "chromatogram" is the chromatography outcome, and "chromatograph" is the chromatography apparatus [19]. Several of the essential aspects of chromatographs include machines designed for molecule isolation termed columns and high-performance compressors for distributing solvents at a steady flow rate, among some of the advanced systems created for chromatography. The technique once known as HPLC was known simply as "LC" as associated technologies were becoming more advanced. Ultrahigh-performance liquid chromatography (UHPLC), that is suitable of incredible examination, is becoming increasingly widely being used now. HPLC could only evaluate chemicals that are immersed in solutions [20]. HPLC isolates chemicals dispersed in a diluted solution, allowing for descriptive and analytical examination of which constituents are exists in the model and how much of every element is present. The nanographene oxide polypyrrole composite (NGPPC) was produced and studied in this study using a simple approach. In order to extract methamphetamine from urine, NGPPC was employed as a DSPE adsorbent. HPLC technique was used to regulate and quantify the amount of MA. The positive relative recuperation analysis indicates that using NGPPC in the DSPE method is a novel sample preparation approach that might be used in effective analysis and medical labs. The remaining sections are arranged as follows. In Section 2, the related work was presented. The materials and methods are in Section 3. Section 4 put the result and discussion to the test in terms of performance and efficiency, with figures and charts displaying the findings. The final section summarises the paper's conclusions.

2. Related Works

A device that extracts amphetamines and methylenedioxyamphetamines from urine is created using a spin column filled using octadecylsilane-bonded monolithic silicon to deal with the challenges of solid-phase extracting. The National Institute of Technology and Evaluation purchased methamphetamine (MA) hydrochloride as well as produced amphetamine (AP) hydrosulfate had been tested for quality. MDMA and MDA (methylenedioxyamphetamine and methylenedioxyamphetamine) have been acquired. The medicines are digested in 0.01 M HCl and kept at 4°C and

refrigerated to make the standard stock solution volumes (1.0 mg/mL). GL Sciences provided the spinning columns. A normal adult provided drug-free urine, which was kept at 20 degrees Celsius until examination. The preactivated columns were filled with urine (0.5 mL), buffer (0.4 mL), and methoxyphenamine (internal standard). During additional permits and washing, the columns were centrifuged (3000 rpm, 5 min). Despite evaporating, the adsorption analytes are subsequently rinsed and examined using high-performance column chromatography. Limit of detection is 0.1 g/ml, linear curves (drug concentrations of 0.2–20 g/mL), and correlation coefficients >0.99. This suggested technique is also not applicable to medicines composed of organic substances, but it is also very repeatable for toxicology testing in urination. Since both specimens and the solvents move in only one direction, there is no chance of sample contamination. Furthermore, since the samples may be recovered with a tiny amount of solvent, this approach has cost expensive [21]. The research offers an extremely specific stir bar sorptive extraction technique for direct estimation of amphetamines in samples taken employing carbon-coated magnetic nanoparticles like a unique stir bar covering. Satisfactory linearity will be reported in the maximum concentration of 20–2000 ng/mL for amphetamine and 20–2500 ng/mL for methamphetamine, including 30–1500 ng/mL for pseudoephedrine using solvent evaporation circumstances. The created recommended approach tested the affirmative urine specimen with successful results. The proposed stir bar sorptive extraction method was used in a variety of forensics as well as medical facilities, according to the findings. The MNC sol gel-coated stir bar demonstrated good reliability and selectivity while determining mixtures in a complicated urine mixture. In comparison to conventional SPME fibers, the experimentally stir bars had a deeper protective coating that resulted in improved removal efficiencies. Additionally, the suggested technique has a modest training procedure, quick sample preparation duration, and is worthwhile. Furthermore, the good recovery observed for the studies given in this paper confirms the suggested technique's usability in the majority of related different laboratories. Given the continuous creation and innovation of various coverings, other obstacles should be solved, such as liquid desorption efficiency, the difficulty of reanalysis following heating desorption, coated condition monitoring after so much usage, and the blending of old and new twisters. SBSE does not have the best precisions (RSD) when compared to certain other extraction processes because stir-bars are costly and should be recycled for numerous extraction processes as much as the covering is in perfect shape [22].

The utility of solid-phase microextraction (SPME) in the perseverance of a growing number of high volatility, as well as semivolatiles samples in biological matrices and components, is being investigated. In spite of the problems posed by minuscule concentration ratios and lengthy absorption coefficient durations, semivolatiles have grown increasingly popular as experimental targets in recent years. Because of these constraints, amphetamines were selected as potential candidates for the semivolatiles category, and evaluation methodologies were devised. Amphetamines are routinely

tested in matrices that are notoriously difficult to analyze. Solid-phase microextraction has proven to be helpful for these types of investigations since it reduces the amount of interaction between the sample and the fiber. The amphetamines were extracted using human urine that used a 100 mm polydimethylsiloxane- (PDMS-) coated SPME fiber. Gas chromatography (GC) with flame-ionization monitoring has been used to determine the presence of amphetamine (FID). To achieve constant separation, temperature, duration, and sodium concentration have been tuned. A straightforward method for testing amphetamine (AMP), methamphetamine (MA), 3,4-methylenedioxyamphetamine (MDA), 3,4-methylenedioxy-N-methamphetamine (MDMA), and 3,4-methylenedioxy-N-ethylamphetamine (M amphetamine (19.5–47%) and methamphetamine (20–38.1%)) had higher recovery rates than MDA (5.1–6.6%) (5.4–9.6 percent). SPME is a fast, solvent-free extraction method that can be used instead of standard liquid-liquid as well as solid-phase extraction for amphetamine detection in organic resources. The focus of this research is to use the HS-SPME to obtain maximum analyte recoveries. As a result, a simple way of determining AMP and MA in urine samples was devised, as well as a distinct approach for determining MDA, MDMA, and MDEA. However, one of the major disadvantages of SPME approaches is the restricted amount of readily accessible column chromatography (fiber materials), which only comprises the polarities range of targeted analytes to a degree [23].

In the subject of systems biology, there must have subsequently become a surge in the rise in the popularity of Rezaee's dispersive liquid-liquid microextraction (DLLME), which was first created in 2006. DLLME is a compact liquid-liquid extraction method with a much lower acceptor-to-donor stage ratio than conventional systems. The use of DLLME in conjunction with various analysis methods including atomic absorption spectrometry (AAS), inductively coupled plasma-optical emission spectrometry (ICP-OES), gas chromatography (GC), and high-performance liquid chromatography (HPLC) for preconcentration and perseverance of synthetic samples in various kinds of materials is discussed. Through the use of an additional solvent to alter the thickness of the extracting mixture, and through the use of ionic liquid-based DLLME to determine synthetic organisms perhaps in the case of excessive sodium content, the systematic study discusses significant breakthroughs in DLLME, such as displacement-DLLME, through the use of an additional solvent to alter the thickness of the extracting mixture. DLLME is an extraction method that was created during the past decade and involves the dispersion of small droplets of extraction solvent in an aqueous sample. This technique was initially used to extract lipids from aqueous samples. A hazy complex is made when a suitable combination of the extractant as well as the disperser liquid with elevated mixtures including both aqueous and organic stages is quickly infused into the acidic suspension of the specimen, and a fine spatter of such removal liquid dissipates in the liquid sample. The small droplets settle at the bottom of the cylindrical glass beaker after centrifugation of the hazy liquid. The solutes have been retrieved out from exact guess and focused on a small capacity of the

deposited process, where they can be determined using standard methodological approaches. Despite DLLME providing exceptional achievement in the liquid solution, it is still not applicable to various matrixes like bioactive molecules. As a result, additional upgrades are required. The utilization of comparatively massive quantities (i.e., mL) of disperser liquids, that process can be broken down the aqueous solubility of specimen matrixes into the extraction solvent stage, is one of the major drawbacks of DLLME [24].

The measurement of methamphetamine as a reference component in biological material was recorded using graphene oxide enhanced two-phase electromembrane extraction (EME) combined with gas chromatography in this research. The inclusion of graphene oxide in the hollow fiber walls could improve the available surface area, chemical contacts, and the polarization of the supporting stream membranes, leading to a rise in sample movement. Comparison research was conducted among graphene oxide and graphene oxide/EME techniques to see how the inclusion of graphene oxide in the supporting liquid membranes affects removal efficiency. The research clearly reveals that immobilizing GO in barriers is an effective way to improve EME performance. This is most likely due to the discovery of a novel channel for bulk transport of METH through the SLM, and as a result, the suggested approach is much more effective and sensitive than traditional EME. The extracting conditions were calculated, including the kind of organic system, supplier stage pH, stirring speed, duration, power, sodium additions, and graphene oxide concentrations. The suggested microextraction approach had a lower detection limit (2.4 ng/mL), significant preconcentration factors (195–198), and a high compared recoveries (95–98.5 percent) within optimal circumstances. Ultimately, the approach was used to properly precautions methamphetamine levels in urinary, and samples were taken. Despite separation without agitating being possible, the GC signals remained substantially weaker than many of those obtained with stimulation. As a result, 1000 rpm was chosen for future research. The mixing rate promotes extracting by increasing convective in the liquid sample. Nevertheless, it is possible that SLM was partially diminished at greater agitation rates, and that organic phase leaking from the SLM occurred [25].

3. Materials and Methods

3.1. Compounds and Reagents. Merck Chemical compounds provided 2-methylimidazole, sodium hydroxide, hydrogen chloride, potassium dihydrogen phosphate, acetonitrile, methanol, and acetone (all HPLC grade) (Darmstadt, Germany). TitraChem provided the zinc nitrate (Tehran, Iran). Sigma-Aldrich provided methamphetamine hydrochloride sample solutions 1000 g/mL in methanol (USA). Milli-Q water system (Darmstadt, Germany) provided ultrapure water.

3.2. Device for Chromatography. During pressure in an oxygen environment with a gold layer (DST1, Nanostructured coatings co., Tehran, Iran), scanned transmission electron (SEM) (MIRA3 FEG-SEM, Tescan, Czech Republic) has been

used to analyze the diameter of the adsorption (MIRA3 FEG-SEM, Tescan, Czech Republic). The Tensor 27 FTIR equipment (Bruker, Germany) was used to acquire their spectroscopy of the specimens (made as KBr disc). On a D5000 (Siemens, Germany) device, powdered X-ray diffraction patterns (XRD) have been acquired. A Zetasizer (Nanotracs Wave, Microtracs, Germany) was used to determine the zeta potential. HPLC assessment was conducted with the use of a Knauer (Germany) machine with a UV-visible detection. As a chromatographic purification column, a C18 column (5 m particle size, 4.6 mm i. d. 25 cm) (Knauer, Germany) has been used at a flow rate of 1 mL/min, and the mobile stage has been composed of acetonitrile-phosphate buffer (10 mM, pH = 3.5) in a 20:80 (V/V) ratio. All test subjects were given the opportunity to give their permission from the participants.

3.3. Preparation of NGPPC Compounds. In a previous paper, nanographene oxides (NGO) were produced by employing an enhanced version of the Hummer process. The modified Hummers method, which is a process that is simple, does not take a lot of time, and does not cost a lot of money, was used to synthesis GO. In addition, utilizing this process results in the introduction of an increased number of hydrophilic groups inside the carbon material, there is no release of hazardous gases, and the conductivity of the material is improved. The following is a summary of the synthesis techniques: 1 gram of graphene was combined with 24 mL of 98 percent H₂SO₄ and teamed inside a frozen bucket. Then, as an oxidation reaction, 3 g KMnO₄ was gently applied. Second, the balloons were placed in an oil tank as well as the temperatures of the reaction were set to 35–40 degrees Celsius. Following that, after approximately 30 minutes of mixing, a light brown colour appeared. The temperature was then increased to 98°C by adding 30 mL H₂O. Mixing was therefore maintained for the next 30 minutes. The brownish brown tint was accomplished by combining 1 mL of H₂O₂ (30%). Dual distilled water and HCl were used to purify and clean the finished version (5 percent). Lastly, water was put into the system and vortexed vigorously to produce a uniform suspension. To generate a nano-GO solution, the mixture was stirred continuously for 40 minutes. Glassware balloons were filled with 50 mL of produced nanographene oxide solution and then stirred for 15 minutes in a nitrogen atmosphere. A total of 0.068 g of pyrrole was combined with the reaction mixture that was then agitated for 20 minutes. The heat of the reaction will therefore be regulated to 0–5°C by utilizing an ice bath continuously stirring for 6 hours, and 20 mL of 2.5 M FeCl₃·6H₂O solutions will be added to the mixture. The resulting dark mixture was centrifuged and then rinsed three times using water and ethanol. The resulting black nanocomposite was placed in the oven to dry at 50°C and used as a DSPE adsorbent [26].

3.4. Acquiring Urine Specimen. The task requires a normal participant to submit a drug-free urine specimen. The specimens were stored at 4°C in a polyethylene container once they were used. MAHAN treatment clinic promptly gathered appropriate urine specimens (Tabriz, Iran). First, 1 g/mL methamphetamine was combined with 5 mL of urine from a healthy subject. Irresolvable small solid particles have

been collected and later detached by centrifuging at 5000 rpm for 10 minutes at ambient temperature after the pH of the urine specimen was corrected to 10. (uni 320, Pole Ideal Tajhiz Co., Iran). For the remainder of the investigation, the supernatant solution was transferred to a fresh container as well as maintained at 4°C [27].

3.5. Method for Dispersive Solid Phase Extraction. The following is how the extracting process has been done: the extract was mixed for 5 minutes after 50 mg of the adsorption was introduced to 6 mL of methamphetamine (0.1 g/mL) spiking urinal. The inclusion of hydrophilic functional groups causes the adsorption to disperse properly in the urine medium, resulting in increased contact between the analyzer and the adsorption. The supernatant was removed after centrifuging the material. To the gathered adsorbent materials, 400 liters of methanol was poured as a desorption solution. The samples were centrifuged following 10 minutes of sonication (30 seconds, Farasout, Iran), and then, 20 liters of the supernatant was fed into the HPLC-UV analysis system.

4. Result and Discussions

4.1. Analysis of Nanographene Oxide Polypyrrole Composites. NGPPC was categorized using Fourier transform infrared spectroscopy (FTIR). The appearance of a spike at 3434 is linked to the hydroxyl group's stretching vibrating band on the NGO porous structure. The carboxylic C=O functional group of NGO is represented by the maximum at 1703 cm⁻¹. In 1634 cm⁻¹, a stretching vibration of C=C emerged. NGPPC's FTIR spectroscopy was investigated. The existence of a spike at 1742 cm⁻¹ is linked to the NGO's C=O structural formula. The peak indicates that NGO has been incorporated into the polymeric nanocomposite composition. C-C, C-N, and N-H stretching vibrations of polypyrrole have reached a maximum at 1550, 1460, and 3442 cm⁻¹. The NGO and NGPPC X-ray diffraction patterns (XRD) were studied. The interplanar separation increases of chemical reduction and remaining unoxidized graphene, correspondingly, were represented by the spikes at $2\theta = 11$ and 26° . The existence of a moderately broad peak at $2\theta = 11.5^\circ$ corresponds to the typical peaking of pyrrole in the NGPPC framework, indicating that pyrrole has been incorporated into the nanographene oxide structure. With just a mean range of 22 nm, the fine complex structures of NGO were clearly visible without any further amorphous structure. The layered structure of NGO is combined with uniformly distributed polypyrrole-covered polymeric on the NGO surfaces including on the NGO sheeting including a mean range of 42 nm, as seen in the SEM image of the NGPPC. The observations of the zeta potential were carried out. The -32.5 mV zeta potential measured demonstrated the existence of strongly negative charging on the NGPPC surfaces, which improved NGPPC interactions with methamphetamine and NGPPC distribution in the medium.

4.2. GO and NGPP Absorption as DSPE. A comprehensive one-factor-at-a-time experimental method will be used to enhance the extracting characteristics. Significant removal

parameters were analyzed, including the quality and number of the extracting solvent system, pH, ionic strength, type and number of adsorption, urine output, mixing speed, extraction time, and desorption time. In a urine sample, the extracting effectiveness of nano-GO and NGPPC in methamphetamine separation were examined. NGPPC had a two-fold better removal efficiency than GO, according to the findings. Following polymerization by pyrrole, the augmentation of phenolic cycles on the NGPPC resulted in the highest association between methamphetamine and NGPPC surface area as shown in Figure 2.

The most effective dosage of NGPPC in methamphetamine removal has been determined through experimenting with new amounts of NGPPC (40-60 mg) in the process of extraction. Once the highest peak regions were reached, 60 mg of NGPPC was administered. The increasing concentration of NGPPC in the maximum absorption wreaked havoc. It could be owing to adsorbent aggregating at greater NGPPC concentrations, which reduces the effectiveness of NGPPC with methamphetamine combinations. Figure 3 shows the graph of the adsorbent's quantity.

4.3. Extraction Solvents and Efficient Volume. The different compounds remained pushed to its limits to see which one was best in removing methamphetamine from NGPPC porous structure. Multiple types (such as methanol, acetonitrile, and acetone) have been tested for this function. Figure 4 depicts the graph of desorption solvents. The results show that methanol is the most efficient solvent for extracting. Methanol has a greater analysis of interactions and is, therefore, more effective in desorbing methamphetamine from the NGPPC surfaces.

It is critical to adjust the effective volume of methanol. As a result, methanol volumes ranging from 300 to 900 liters have been investigated. A capacity of 300 liters is sufficient to desorb the greatest quantity of methamphetamine from the surfaces of the NGPPC. As a consequence of analyte diluting in the medium, the steady increase of desorption solvents exhibited a reduction. Figure 5 shows the quantity graph of desorption solvents.

4.4. The Efficiency of Urine Volume, pH, and Ionic Strength. An additional factor impacting removal efficiency is urine quantity. As a result, urine quantities ranging from 1 to 8 mL have been examined. The findings demonstrate how utilizing 5 mL of urine resulted in the highest removal effectiveness. The adsorbent's surface energy is a factor that influences removal efficiency and adsorbent aggregation. The pHs of the solution have been studied in the series of 4–12. The quantity of negative controls on the NGPPC increases as its pH of the medium rises from 4 to 10, resulting in a better connection between the positively charged samples as well as negatively charged chemical adsorption. Figure 6 depicts the graph for urine specimen quantity.

At a pH of 10, the behaviour achieves its pinnacle. The removal rate was reduced when pH = 12 was used. Given that the pKa of methamphetamine is 10.1, it changes back to the normal analyzer at higher pH levels. This has the impact of limiting the effective interactions between the

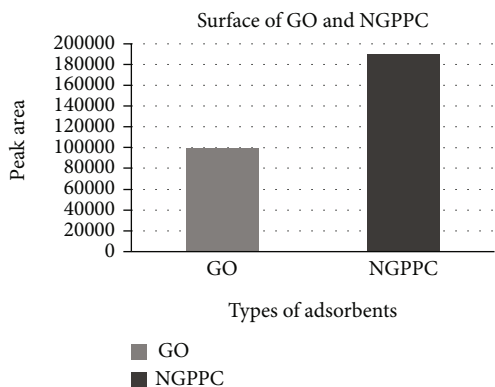


FIGURE 2: Surface area graph of GO and NGPPC adsorbents.

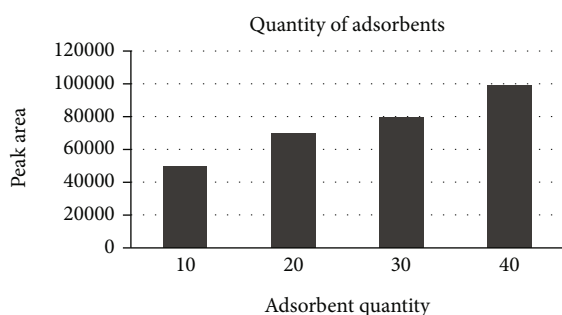


FIGURE 3: Graph of adsorbent's quantity.

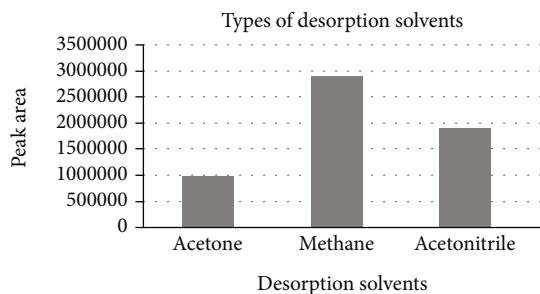


FIGURE 4: Graph of desorption solvents.

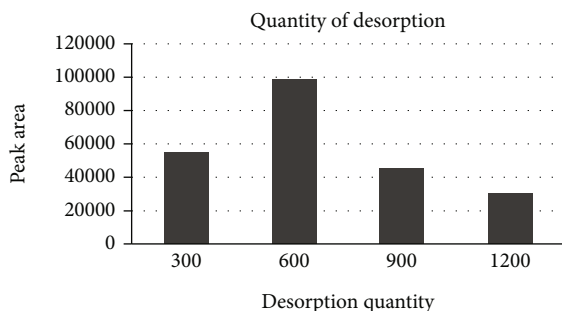


FIGURE 5: Graph of desorption solvent's quantity.

analyzer and the adsorption of negative charges. It can be assumed from this fact that the cation form of this molecule will predominate almost totally in the natural environment.

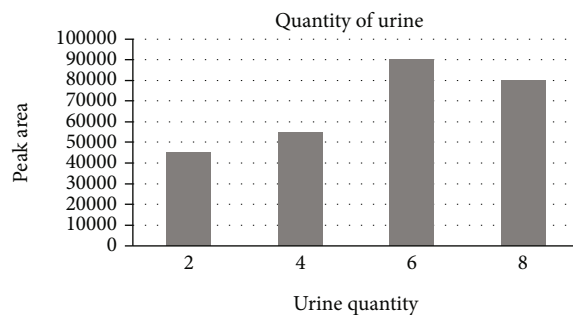


FIGURE 6: Graph of urine quantity.

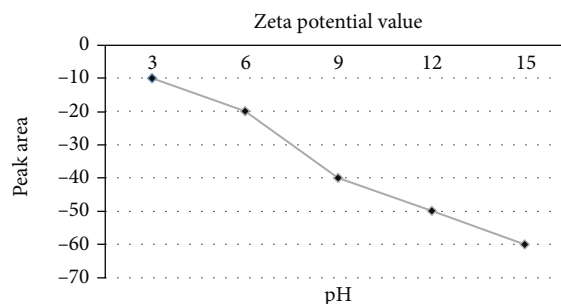


FIGURE 7: Zeta potential value of pH.

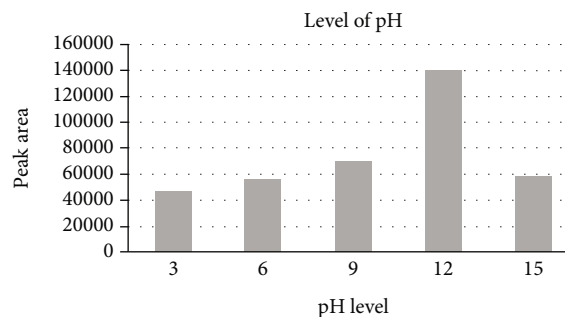


FIGURE 8: Graph of pH level.

As an outcome, pH = 10 has been selected as the best extracting pH for the separation method.

Plotting zeta potential as a proportion of pH could clarify such behaviour as shown in Figure 7. As can be seen, increasing the pH between 4 and 10 results in a greater number of adverse charging on the NGPPC adsorption, resulting in maximal analysis separation.

Figure 8 shows the graph of pH level. Despite the fact that a continual improvement in pH up to 12 resulted in an increment in negative controls on the NGPCC adsorbent, the removal efficiencies remained unchanged. The outcome is connected to the usual method of methamphetamine, which has a pK_a of 11.1 in a pH of 12, resulting in lower removal efficiencies. By applying 0–7% (W/V) NaCl towards the extracting solvent, the impact of ionic strength has been examined. The research was conducted without putting salt since the removal efficiencies did not vary significantly (data not shown).

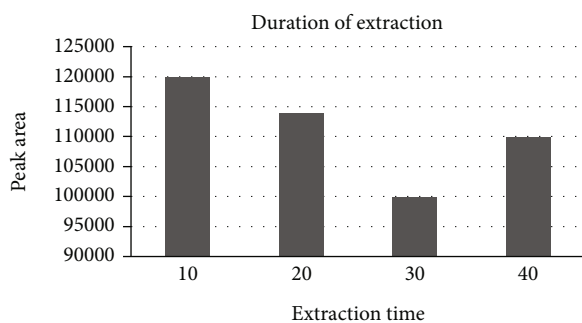


FIGURE 9: Graph of extraction duration.

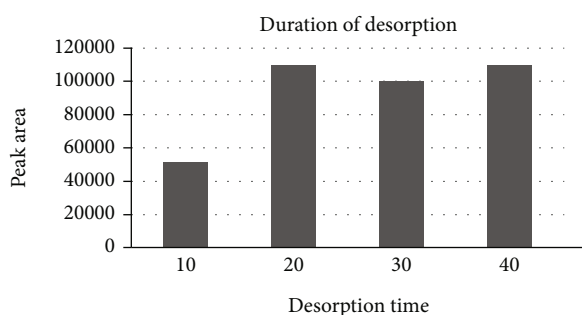


FIGURE 10: Graph of desorption duration.

4.5. Extraction and Desorption Duration and Mixing Rate. Establishing equilibrium in extracting processes is critical, and duration is a changeable variable to accomplish so. The procedures were tested over a time span of 10–40 minutes, with peak removal efficiencies of 10 minutes as shown in Figure 9. Because the process has remained stable, increasing the extracting duration has no effect on the removal efficiencies.

Desorption duration had also been examined in the 10–40 minute range as depicted in Figure 10. The equilibrium was reached in ten minutes, and a gradual increase in desorption duration had no effect on removal efficiencies. Furthermore, mixing speed influences the development of the NGPPC-methamphetamine interactions.

As a result, a stirring frequency of 2000–8000 was calculated as shown in Figure 11. The aggregate of NGPPC is much more likely at greater mixing speeds. Furthermore, the process reaches equilibrium at 6000 rpm, and greater mixing rates have little effect on the methamphetamine maximum absorption.

4.6. Adsorbent Capability and Reusability. From a cost standpoint, the adsorbent's application programs were critical. As a result, the adsorbent's reusability was evaluated using extraction efficiency conditions as depicted in Figure 12. The outcomes indicated that higher to 6 times reusing of the produced adsorbents, there was no substantial shift in removal efficiencies. The following formula has been used to compute the adsorbent capability in the following equation.

$$E_q = \left(\frac{(b_1 - b_2)u_v}{a_m} \right) \times 100. \quad (1)$$

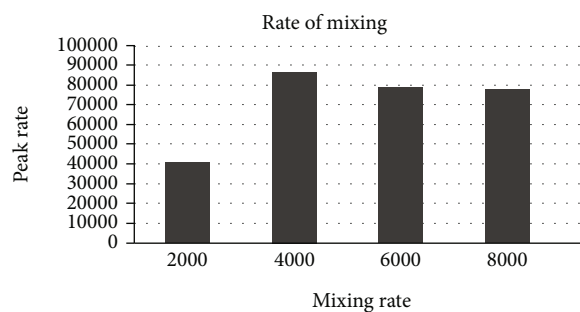


FIGURE 11: Rate of mixing.

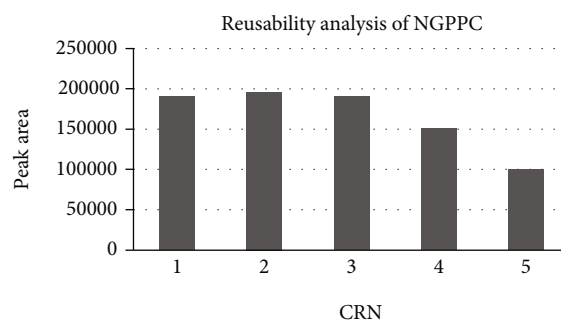


FIGURE 12: Analyzing NGPPC reusability.

The preliminary and equilibrium quantities of the MA in the urine specimen (g/mL) are b_1 and b_2 , correspondingly. a_m is the mass of adsorption, while u_v is the capacity of urine (mL) (g). The adsorbing efficiency measured was 0.3 mg/g.

4.7. Verification of the Methodology. For demonstrating the technique's suitability for extracting methamphetamine through the urinary medium, certain analysis features of the proposed technique were established. Limit of quantification (LOQ), relative standard deviation (RSD), limits of detection (LOD), coefficient of determination (CO), and linearity have been investigated in three repetitions with five concentrations that covered the calibration graph. Table 1 shows the properties of DSPE analysis for MA.

In the range of concentrations of 50–2500 ng/mL, the technique remained linear. When comparing the suggested method to certain other recently reported approaches, it was discovered that DSPE-derived adsorbents remove pharmaceuticals from biological materials having acceptable and repeatable outcomes due to their great physical elasticity, hydrophilic or hydrophobic characteristics, and configurable pore scope as shown in Table 2 and Figure 13.

The UV detector's experimental restriction, as opposed to fluorescence, mass spectroscopy, or gas chromatography, may account for the relatively greater concentration range. In three different concentrations comprising the calibration graph, Table 3 shows the investigative sensitivity and specificity of the suggested novel DSPE technique. The findings supported the product's repeatability and efficiency.

4.8. Analyzing Real Samples Using the DSPE Approach. Real samples were tested to demonstrate the usability of the established DSPE approach. As a result, the addicts' urine

TABLE 1: Properties of DSPE analysis for MA.

| Sample | CR | CO | Limit of detection | Limit of quantification | Relative standard deviation |
|--------|---------|--------|--------------------|-------------------------|-----------------------------|
| MA | 40-2400 | 0.9945 | 12 | 40.90 | 5.40 |

TABLE 2: Comparison table of proposed and existing methods.

| Sample | Technique | Limit of detection | Recovery |
|-----------------|------------------------------|--------------------|----------|
| Methamphetamine | Solid phase extraction | 100 | 60.6 |
| | Stir bar sorptive extraction | 60 | 82.8 |
| | Solid phase microextraction | 30 | 96.7 |
| | Dispersive liquid-liquid ME | 20 | 98.5 |
| | Proposed-diffusive SPE | 10 | 99.2 |

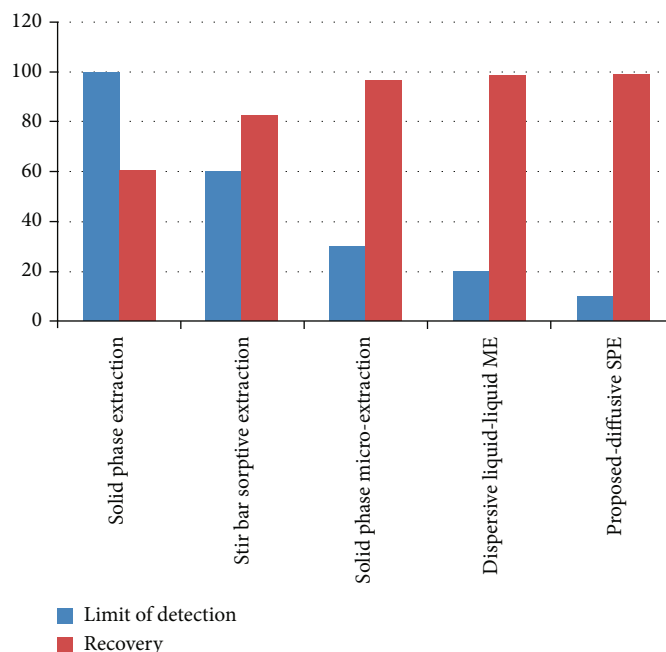


FIGURE 13: Limit of detection and recovery of various models.

TABLE 3: Verification outcomes for the provided DSPE method.

| MA solutions (millilitre) | Within the days | | Between days | |
|------------------------------|-----------------|------|--------------|------|
| | Acc | Sen | Acc | Sen |
| 0.08 | 2.24 | 0.74 | 3.27 | 0.96 |
| 0.2 | 2.19 | 0.52 | 3.26 | 0.82 |
| 1.10 | 5.10 | 0.92 | 6.23 | 0.92 |

extraction was done using NGPPC absorbent and inserted into the HPLC. Whenever positive urine specimens have been analyzed, the presented procedure yielded satisfactory findings, with a comparative recovery efficiency of 99.76 percent. The optimum condition is for current data to be verified using certified reference materials (CRM). Nevertheless, the current data can be trusted since (a) the high-performance liquid chromatography (HPLC) method can qualitatively and quantitatively analyze what components and how much of each component are present in a sample by separating chemicals that have been dissolved in a liquid.

The gold standard technique for separating in the evaluation of whether or not the limited chromatograms is well-matched with variable absorptions of requirements is the HPLC-UV method; and (b) the comparative recovery findings have been adjusted to account for variations in the spiking drug. Concurrent spikes of 0.1 gmL^{-1} methamphetamines, as well as many associated metabolites, were being used to test the selectivity of the innovative DSPE approach in methamphetamine separation. The findings revealed there were no other substantial spikes that might lead to FP results. The procedure is called selectivity, and it is used to extract methamphetamine from urine. The following equation was used to investigate the matrix effect in the following equation.

$$m_e = \frac{Y}{X} \times 100. \quad (2)$$

Assume X as the peak area of the aqueous mixture, as well as Y as the peak area of the postextraction solvent.

The matrix effects were estimated to be 111 percent, indicating that the matrix impact is dismissible.

5. Conclusion

NGPPC was being used to recover methamphetamine through urine medium with high efficiency. The NGPPC synthesizing procedure was easy, and the extraction technique demonstrated good repeatability. Furthermore, the suggested method's medical usefulness was demonstrated by its quick extracting and desorption durations, selectivity, and capacity sample analysis. The suggested method was validated, as well as the coefficient of correlation is of 0.996 indicated that the technique was linear in the absorption in the range contained by the calibration curve (30-800 g/mL). Furthermore, the suggested DSPE approach for methamphetamine identification demonstrated good precision, accuracy, and durability. Even without the involvement of metabolites, methamphetamine might well be recognized. Furthermore, the suggested DSPE approach for methamphetamine identification demonstrated great precision, accuracy, and robustness. The DSPE-based analytical approach has been suggested for specific methamphetamine measurement in biological urine medium with excellent removal efficiencies and a lower detection limit. Furthermore, the approach is quick and inexpensive, with significant recovery efficiency, making it an ideal analysis technique for clinical and forensic laboratories. Methamphetamine may be identified without any metabolite influence. Moreover, the practical and efficient synthesis process stimulates the use of carbon-based compounds in various extraction procedures.

Data Availability

The data used to support the findings of this study are included within the article. Further data or information is available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

The authors appreciate the supports from Wolaita Sodo University, Ethiopia for the research and preparation of the manuscript. This work was funded by the Researchers Supporting Project Number (RSP2023R429) King Saud University, Riyadh, Saudi Arabia.

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