

Magnetic solid-phase extraction method in sample pretreatment

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For improving the crop yield and enhancing the quality of crop production by controlling crop pests and reducing weeds various pesticides are used. Due to excessive use of pesticides in agriculture, they cause extensive pollution to the environment and also risk the human health. From the past few years various pesticides like organophosphorus (OPs), pyrethroid (PYR), triazole have been widely used and therefore their traces may be present and found in our environment, including water, soil, agricultural products, and food . These traces found to be very toxic for people as well as for animals and cause harmful effects on the environment. Therefore, introducing a simple, fast and sensitive method for the removal of pesticides is very desirable.

Sample preparation for the effective extraction of the analytes from a sample matrix is a necessary and important step in the whole analytical process. In recent years, magnetic solid-phase extraction (MSPE) as a relatively new mode of solid-phase extraction (SPE) has received much attention in sample pretreatment. MSPE is based on the use of magnetic or magnetizable adsorbents with high adsorption ability and superparamagnetism. Unlike the traditional SPE, the magnetic adsorbents do not need to be packed into a SPE cartridge but are dispersed in a sample solution or suspension. The adsorbent can be readily isolated from the sample solution with a magnet and the column blocking problems during extraction can be avoided. In addition, it can facilitate the mass transfer of the analytes by drastically increasing the contact area between the solid adsorbent and sample solution, thus reducing the extraction time.

In MSPE, an appropriate magnetic adsorbent is crucial for the efficient extraction of the analytes. Magnetic nano-sorbents have been proved to be <https://assignbuster.com/magnetic-solid-phase-extraction-method-in-sample-pretreatment/>

especially effective as a result of their high specific surface area and highly active surface sites.

Carbon nanostructured materials, due to their large surface area and excellent adsorption capacity, have been successfully used as the MSPE adsorbent for the extraction of some organic compounds. Graphene oxide (GO), also called graphite oxide sheet, is a two dimensional nanomaterial prepared from chemical oxidation of natural graphite. GO can be well-dispersed in water due to its abundant hydrophilic groups, such as hydroxyl, epoxide and carboxylic groups, on its surface.

Furthermore, GO has emerged as a precursor offering the potential of cost-effective and large-scale production of graphene-based materials. Graphene (G), an atomically thin honeycomb lattice of carbon atoms, has received a tremendous amount of research attention in recent years because of its large surface area (theoretical value of $2360 \text{ m}^2 \text{ g}^{-1}$), chemical stability and high electrical conductivity. In contrast, G is an electron-rich, hydrophobic nanomaterial with a large delocalized π -electron system. This endows G with a strong affinity for carbon-based ring structures, which could possibly allow its use as a good adsorbent. In recent years, G has been used as the adsorbent in solid-phase extraction (SPE), solid-phase microextraction (SPME), matrix solid-phase dispersion (MSPD), micro-solid-phase extraction (μ -SPE) and magnetic solid-phase extraction (MSPE).

The introduction of magnetic properties into G could combine the high adsorption capacity of G with the separation convenience of the magnetic materials. A few magnetic G-based materials have been prepared for the

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removal or enrichment of some contaminants from different samples. For example, Wu et al. prepared a G-based magnetic nanocomposite by the in situ chemical coprecipitation method and used it for the extraction of carbamate pesticides from environmental water samples. A Fe₃O₄/graphene oxide (GO) nanocomposite has been synthesized by an electrostatic self-assembly approach and used for the preconcentration of polycyclic aromatic hydrocarbons from environmental samples. Shi et al. have fabricated magnetic G by a simple hydrothermal reaction and used it for the adsorption of small molecules. However, there have been some problems when these composites were used as adsorbents. The Fe₃O₄ nanoparticles (NPs) were attached to a G layer only by physical adsorption or electrostatic interaction, and they may be easily leached out from the G sheets during application. The magnetic Fe₃O₄ particles were exposed and might be oxidized and therefore could lose their magnetism under harsh experimental conditions and repeated use. To solve the above problems, a suitable protective coating on a magnetic core could be used and G could be confined on the surface of the magnetic microspheres. Liu et al. have assembled G on the amino-functional silica-coated Fe₃O₄ core-shell NPs to realize the enrichment of some proteins/peptides. Our group has fabricated a silica-coated magnetic G composite using surface grafting by G (Fe₃O₄@SiO₂-G) via a chemical bonding method. The results indicated that the G bonded on the surface of the magnetic microspheres endows the material with a high adsorption capacity and it is more stable because the silica shell could provide good protection for the Fe₃O₄ core.

So far, the reported G-based magnetic adsorbents have been mainly applied to the extraction of some analytes from water samples, and the reports about their use for more complex sample matrices have been very few. In this paper, Fe₃O₄@SiO₂-G, which was fabricated in our lab by the chemical bonding of G onto the surface of silica-coated Fe₃O₄ NPs, was further explored for the extraction of the fourteen pesticides (diazinon, fenitrothion, chlorpyrifos, parathion, procymidone, ditalimfos, hexaconazole, myclobutanil, kresoxim-methyl, tebuconazole, fenpropathrin, cyhalothrin, cypermethrin, and fenvalerate) in tomato and rape samples. After the preconcentration with the adsorbent, the analytes were analyzed by gas chromatography-mass spectrometry (GC-MS). Several experimental parameters that could affect the extraction efficiencies such as the amount of Fe₃O₄@SiO₂-G, extraction time, sample solution pH, salt concentration, and desorption conditions were studied. The results indicated that the developed method was simple and effective for the determination of the pesticides in the vegetables with good sensitivity and reproducibility.