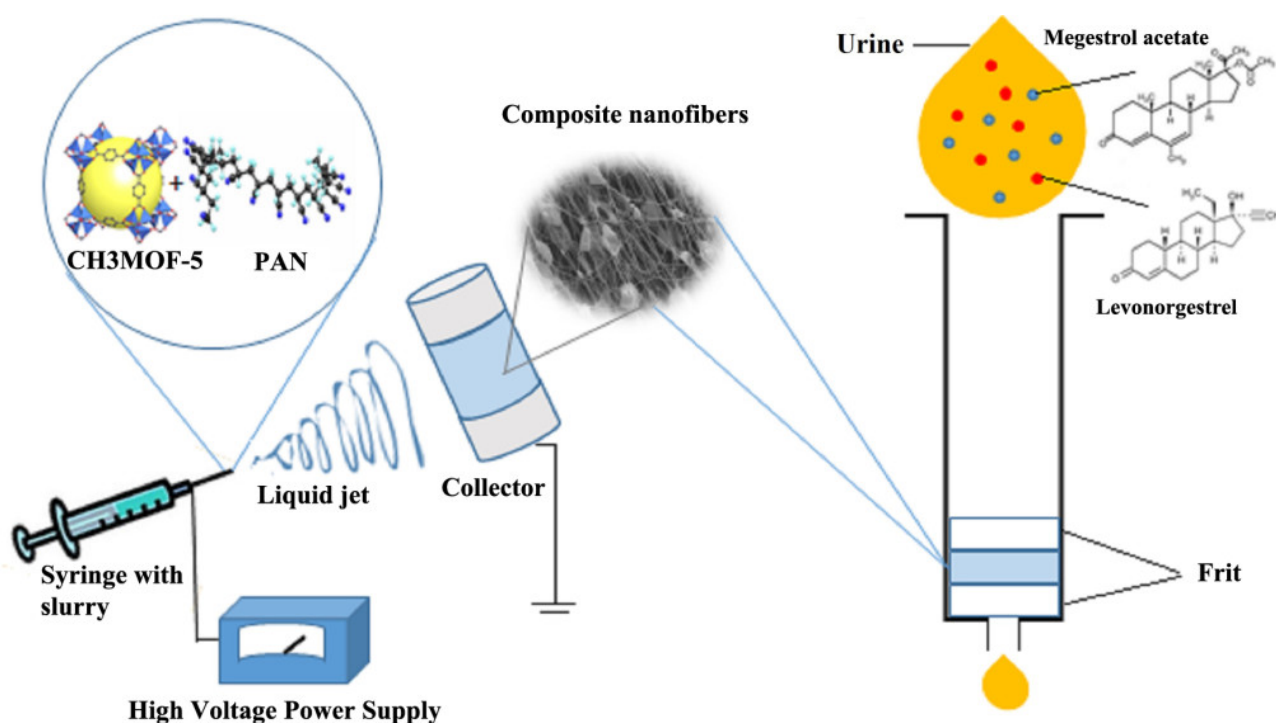


Electrospun CH3MOF-5/PAN composite nanofibers for SPE of levonorgestrel and megestrol acetate

Electrospinning is a very simple, versatile and high-throughput method for generating continuous nanofibers from a wide range of materials including polymers, composites and ceramics with controllable diameters, compositions and morphologies. It was observed that the electrospun nanofibers possess a great potential to serve as a good sorbent material for SPE-based techniques. The main advantage of electrospun nanofibers is their large surface area to volume ratio, which allows using small amounts of the sorbent in the SPE procedure. It reduces the volume of eluting solvents and increases the extraction efficiency for the trace analyses. To date, most of the electrospun nanofibers used for SPE have been polystyrene or nylon 6 polymers and the application of other electrospun nanofibers in sample preparation has been less explored.



Metal-organic frameworks (MOFs) or porous coordination polymers are a new class of crystalline porous materials that have been first introduced by Yaghi and co-workers. The advantages of MOFs as compared to the conventional porous materials, like zeolites or activated carbons, are their extraordinary surface area, microporosity, remarkable low density and ability to be easily designed or modified to have different pore sizes. These advantages make MOFs attractive for the analytical applications. Recently, MOFs were used for SPE, micro-SPE and SPME of some organic and inorganic analytes. Perhaps the most famous MOF ever used is MOF-5. MOF-5 with its high surface area and great porosity has been widely used for gas adsorption studies and molecular simulation analysis. However, problems arise when it is either applied for adsorption in aqueous

matrices or exposed to the low amounts of moistures. This is important because generally water resistance is an important property for a SPE adsorbent in aqueous media while the mobile phase and most of the environmental and biological samples are in the aqueous phase. Recently, researchers have found that it is possible to obtain water-stable MOFs by introducing hydrophobic functional groups into their framework structures. In 2011, Yang et al. synthesized the methyl modified MOF-5 (CH₃MOF-5) analogs using a solvothermal method. It was confirmed that the incorporation of hydrophobic methyl groups into the structure of MOF-5 improved its water resistance.

The present study focuses on the use of CH₃MOF-5/PAN composite for SPE of levonorgestrel (LeV) and megestrol acetate (MA) as the model compounds. They are the synthetic progestational hormones. LeV is widely used in pregnancy prevention in humans. MA is used mainly to treat breast, endometrial, and prostate cancers. The presence of different functional groups in the chemical structures of the target analytes can possibly lead to different interactions such as π-π interactions, hydrogen bonding and hydrophobic interactions between them and the sorbent. On the other hand, due to the remarkable water stability of CH₃MOF-5, it is distinguished from the other moisture sensitive MOFs. Therefore, it can be used as a suitable sorbent for the extraction of the analytes from the aqueous media such as biological fluids.

The nanofibers were packed into the mini-disc cartridges to be used as SPE devices. Under the optimized conditions, the linearity varied in range of 0.05–100 µg L⁻¹ with R² values higher than 0.999. The limit of detection for both of the analytes was 0.02 µg L⁻¹. The applicability of the method was examined by analyzing the analytes in the urine samples. The recovery of the analytes varied in the range of 82.8–94.8% which shows capability of the method for the determination of the drugs in the urine samples.

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[Preparation of water stable methyl-modified metal-organic framework-5/polyacrylonitrile composite nanofibers via electrospinning and their application for solid-phase extraction of two estrogenic drugs in urine samples.](#)

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