Sensitive HPLC Determination of Duloxetine after Extraction Using Magnetic Multi-walled Carbon Nanotubes

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Abstract: Duloxetine, which is used to treat major depressive disorder and diabetic peripheral neuropathic pain, can be analyzed with different analytical methods, including high performance liquid chromatography-tandem mass spectrometry, electrophoresis with fluorescence detection, HPLC with fluorescence detection and so on. However, these methods need time consuming sample preparations or special and expensive instruments. It is necessary to develop a simple, rapid, sensitive and cost-effective method for the determination of duloxetine in biological fluids.

Methods: A combination of magnetic dispersive solid-phase extraction procedure with high performance liquid chromatographic-ultraviolet detection (HPLC-UV) was developed for the determination of duloxetine in human plasma and urine samples. Magnetic Fe3O4 nanoparticles grafted oxidized-multiwalled carbon nanotube (MNPs-MWCNTs) was used as an efficient adsorbent during the preconcentration step.

Results: Under the optimized conditions, the calibration curve was linear over the concentration range of 10-2500 ng mL-1 (R2 = 0.9991). Validation experiments revealed that the optimized method has high percent recovery (96%), high enrichment factor (120), good precision as RSD% (intra-day: 3.28, 3.44; inter-day: 4.29, 4.51 for urine and plasma, respectively, C=1000 ng mL-1, n=6) and low detection limit (2.1 ng mL-1).

Conclusion: Obtained results indicated that the proposed method has advantages of convenience, good sensitivity and high efficiency, and it was feasible for the determination of duloxetine in biological fluids.

Keywords: Magnetic multi-walled carbon nanotubes, magnetic dispersive solid phase extraction, HPLC, duloxetine, urine, plasma.

1. INTRODUCTION

Duloxetine is a selective serotonin and norepinephrine reuptake inhibitor (SNRI) approved to treat major depressive disorder and diabetic peripheral neuropathic pain in the US and Europe [1, 2]. Its solid oral dosage form is usually available as capsules which contain 20, 30 or 60 mg of active constituent in enteric-coated pellets [3].

Numerous analytical methods have been reported for the determination of duloxetine in different matrices including high performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) [2, 4, 5], capillary electrophoresis with fluorescence detection (CE-FL) [6], HPLC with fluorescence detection (HPLC-FL) [7] and reverse phase ultra-performance liquid chromatography (UPLC) [8]. However, HPLC-FL requires analyte derivatization, which is a complicated and time-consuming procedure and other HPLC-MS/MS, UPLC and CE-FL methods require very expensive instrumentation, which are not always readily available in clinical analysis laboratories.

In the past decades, solid-phase extraction (SPE) has considerable attention as a sample preparation technique for the analysis of environmental, food and biomedical analytes [9]. However, traditional SPE is laborious and time-consuming due to the limited rate of diffusion and mass transfer of analytes in the bulk sorbent phases packed in a cartridge [10]. Furthermore, solid particles which may exist in real samples can cause a blockage of cartridges leading to extraction failure. Magnetic dispersive solid-phase extraction (MDSPE) is a new type of SPE, which is based on the adsorption of analytes on micro- or nano-scale magnetic particles [11]. There is no need to be packed the adsorbent into the cartridges in this technique because the particles are homogeneously dispersed in the sample solution during the extraction procedure. In such a dispersive mode, MDSPE not only enhances the extraction efficiency by increasing interfacial area between the solid adsorbent and sample solution, but also overcomes the problems accompany with traditional SPE which mentioned above. Another interesting property of