



ORIGINAL ARTICLE

Trace analysis of mefenamic acid in human serum and pharmaceutical wastewater samples after pre-concentration with Ni–Al layered double hydroxide nano-particles



Hossein Abdolmohammad-Zadeh^{a,*}, Fatemeh Morshedzadeh^b,
Elaheh Rahimpour^a

^aDepartment of Chemistry, Faculty of Sciences, Azarbaijan Shahid Madani University,
35 Km Tabriz-Maragheh Road, P.O. Box 53714-161, Tabriz, Iran

^bDepartment of Chemistry, Payame Nour University of Tabriz, P.O. Box 19395-3697, Tabriz, Iran

Received 1 January 2014; revised 12 April 2014; accepted 25 April 2014
Available online 23 May 2014

KEYWORDS

Mefenamic acid;
Solid phase extraction;
Nano-sorbent;
Nickel-aluminum layered
double hydroxide;
Ultraviolet spectroscopy

Abstract In this work, the nickel–aluminum layered double hydroxide (Ni–Al LDH) with nitrate interlayer anion was synthesized and used as a solid phase extraction sorbent for the selective separation and pre-concentration of mefenamic acid prior to quantification by UV detection at $\lambda_{\max} = 286$ nm. Extraction procedure is based on the adsorption of mefenamate anions on the Ni–Al(NO₃⁻) LDH and/or their exchange with LDH interlayer NO₃⁻ anions. The effects of several parameters such as cations and interlayer anions type in LDH structure, pH, sample flow rate, elution conditions, amount of nano-sorbent and co-existing ions on the extraction were investigated and optimized. Under the optimum conditions, the calibration graph was linear within the range of 2–1000 µg/L with a correlation coefficient of 0.9995. The limit of detection and relative standard deviation were 0.6 µg/L and 0.84% (30 µg/L, $n=6$), respectively. The presented method was successfully applied to determine of mefenamic acid in human serum and pharmaceutical wastewater samples.

© 2014 Xi'an Jiaotong University. Production and hosting by Elsevier B.V. All rights reserved.

1. Introduction

Mefenamic acid (MFA, 2-(2,3-dimethyl phenyl) aminobenzoic acid) is a prevailing non-steroidal anti-inflammatory drug which is used as a potent analgesic and anti-inflammatory agent in the treatment of several pathologies such as osteoarthritis, nonarticular rheumatism, sport injuries, and other painful musculoskeletal illnesses

*Corresponding author. Tel.: +98 4124327500; fax: +98 4124327541.

E-mail addresses: h.abdol@azaruniv.edu,

h_abdol@yahoo.com (H. Abdolmohammad-Zadeh).

Peer review under responsibility of Xi'an Jiaotong University.

[1,2]. Overdose of mefenamic acid produces toxic metabolite accumulation that causes nausea, vomiting and occasionally bloody diarrhea [1]. On the other hand, MFA is a diphenylamine derivative pollutant and the third compound on the European Union list of priority pollutants [3]. Many studies have revealed that MFA cannot be effectively removed by conventional sewage treatment plants and that it has been detected at trace level in the effluent of wastewater treatment plants [4–7]. Due to the vital importance and widespread use of MFA, the need for the development of simple and sensitive analytical methods for trace analysis of drug is increasing.

So far, various analytical methods regarding MFA determination in pharmaceutical formulations and biological fluids have been published in literature. Some reported methods are spectrophotometry [8–11], fluorimetry [12–14], potentiometry [15–18], chromatography [19–23], chemiluminescence [24,25] and capillary electromigration [26,27]. However, to determine the trace levels of drug, spectrophotometry may be used, especially in combination with extraction for separation of special purpose component from the main admixture. Solid-phase extraction (SPE) techniques have recently been among the most popular separation methods for the enrichment of analytes prior to their determination. The basic principle of SPE is the pre-concentration and purification of analytes from solution by sorption on a solid sorbent [28]. SPE has several advantages over other techniques, such as low cost, low consumption of organic solvents, high enrichment factor, high recovery, safety with respect to hazardous samples and the ability of combination with different detection techniques in the form of on-line or off-line mode [29]. Recently, nano-meter sized materials have been used as sorbents in SPE procedures.

Layered double hydroxides (LDHs) are a class of synthetic ionic inorganic compounds with a similar structure to clays and with the general formula of $[M_1^{2+}_x M_2^{3+}_y (\text{OH})_2]^{x+y} [A^{n-}]_z \cdot m\text{H}_2\text{O}]^{x-y-z}$, where M^{2+} is a divalent metal ion like Zn, Mg, Cu, Co or Ni, M^{3+} is a trivalent metal ion like Al, Fe or Cr, x is the ratio of $M^{3+}/(M^{2+}+M^{3+})$ and A^{n-} is a n -valent anion [30]. The layer structure of LDHs is based on that of brucite $[\text{Mg}(\text{OH})_2]$, which is typically associated with small polarizing cations and polarizable anions. Their structure is based on a series of layers, where a divalent metal cation is located in the center of octahedron, and two-dimensional infinite layers are formed by edge-sharing of octahedral. The partial substitution of divalent cations by trivalent ones generates a positive charge on the layers that is balanced by

anions or molecules of solvent. Interlayer anions can be exchanged with various kinds of inorganic or organic anions by ion exchange reaction or surface adsorption [31,32]. Scheme 1 shows ion exchange mechanism of mefenamate anions, as organic anions, with the interlayer anions in a LDH structure.

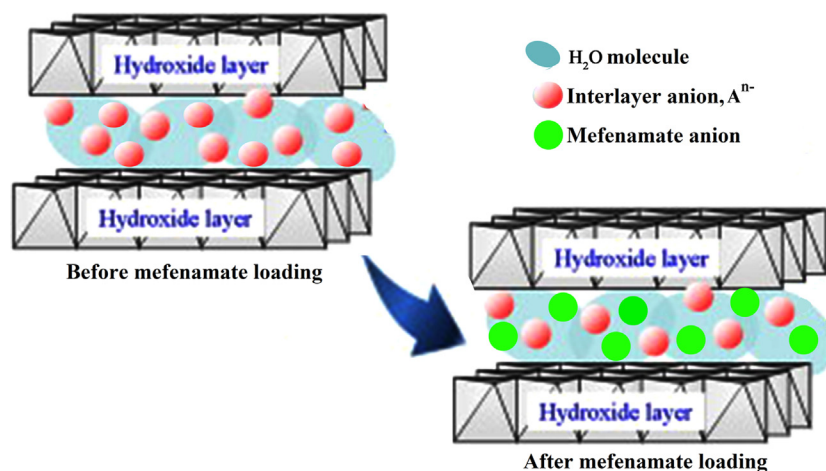
In the present work, a simple SPE system based on Ni–Al(NO_3) LDH was developed for the separation and pre-concentration of MFA prior to determination by spectrophotometry. To the best of knowledge, there is no report concerning the application of the LDHs in SPE of MFA. The effect of various experimental parameters on the extraction efficiency of MFA was investigated and the presented method was successfully used for trace analysis of MFA in various real samples.

2. Experimental

2.1. Apparatus and instruments

The UV–vis absorption spectra and intensity measurements are recorded on a 1601 PC UV–vis spectrophotometer (Shimadzu, Japan). A 2 mL polypropylene cartridge (30 mm × 7 mm i.d.) (Shafa Co., Iran) containing 250 mg of Ni–Al(NO_3) LDH with cotton-fitted ends is used for the extraction of the analyte. A vacuum pump model DV-85N-250 (Platinum Co., USA) is used for controlling the flow rate of solution throughout the column.

In order to obtain better insight into the structural properties of LDH, XRD data were collected on a Bruker-D8 advance X-ray powder diffractometer using $\text{Cu}_{K\alpha}$ radiation source ($\lambda = 0.154$ nm) operating at 40 kV and 30 mA. The patterns were recorded at 2θ from 2° to 70° at room temperature. Also, Fourier transform infrared (FT-IR) spectra (4000 – 400 cm^{-1}) were recorded using a Shimadzu FT-IR Spectrometer, model 8400 (Japan). The samples were mixed with KBr with a sample/KBr weight ratio of 1/100 and pressed into a disk. Morphological characterization of the synthesized Ni–Al(NO_3) LDH was performed using a scanning electron microscope (SEM) model Hitachi S 4160 and a transmission electron microscope (TEM) model PHILIPS SM10. A centrifuge (Shimifann CE. 86) with a relative centrifugal force of 2810g (4000 rpm) was used to accelerate the phase separation. The pH was adjusted using a Motrohm pH-meter (model 827, Switzerland) with a precision of ± 0.01 . An electrical furnace (Exciton Co.,



Scheme 1 Ion exchange mechanism of mefenamate anions with the interlayer anions in a LDH structure.

Download English Version:

<https://daneshyari.com/en/article/2507720>

Download Persian Version:

<https://daneshyari.com/article/2507720>

[Daneshyari.com](https://daneshyari.com)