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RESEARCH ARTICLE

Bioassay studies of Risperidone and its active metabolite in rat dried blood spots and dried plasma spots using LC-ESI-MS/MS: Comparison of their pharmacokinetic profiles

Gangu Naidu Challa, Narendra Varma Nimmu, Ramachandra Bondigalla, Manikanta Swamy Arnipalli

First published: 02 August 2020
https://doi.org/10.1002/sscp.202000021

Abstract

A highly sensitive and specific liquid chromatography-elctrospray ionization tandem mass spectrometry method was developed and validated for rapid determination of risperidone and its active metabolite in rat dried blood spots and dried plasma spots. Chromatographic separation was achieved through ZIC®-HILIC (250 × 4.6 mm, 5 µm) at 25°C using 10 mM ammonium acetate (pH: 3.0, adjusted with acetic acid) and acetonitrile (12:88, v/v) as a mobile phase. Tandem mass spectrometry detection was performed in positive ion electrospray ionization using multiple reaction monitoring mode to monitor precursor → product ion transitions *m/z* 411.2→191.1 for Risperidone, *m/z* 427.2→207.1 for its metabolite (Risperidone-9-OH), respectively. The lower limit o quantification of Risperidone and Risperidone-9-OH were 0.95 and 0.90 ng/mL in rat dried blood spots, respectively. The developed bioassay method was successfully applied to study the pharmacokinetic profile of Risperidone and Risperidone-9-OH using Iloperidone as an internal standard. As per pharmacokinetic studies, C_{max} of Risperidone and Risperidone-9-OH in dried blood spots was 36.2 and 28.9 ng/mL; and in dried plasma spots was 38.2 and 30.2 ng/mL, respectively. Similarly, t_{max} of Risperidone and Risperidone-9-OH in dried blood spots was 1.4 and 14.7 h; and in dried plasma spots was 1.54 and 15.4 h, respectively.

CONFLICT OF INTEREST

The authors have declared no conflict of interest.

Supporting Information

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SEPARATION SCIENCE PLUS / Volume 3, Issue 10

INSIDE FRONT COVER | Free Access

Front Cover: Bioassay studies of Risperidone and its active metabolite in rat dried blood spots and dried plasma spots using LC-ESI-MS/MS: Comparison of their pharmacokinetic profiles

Gangu Naidu Challa, Narendra Varma Nimmu, Ramachandra Bondigalla, Manikanta Swamy Arnipalli

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Graphical Abstract

DOI: **10.1002/sscp.202000021**

The cover picture shows a highly sensitive and specific LC-ESI-MS/MS method was developed and validated for the bioassay of Risperidone (RISP) and its active metabolite Risperidone-9-OH (RISP-9-OH) in rat dried blood spots (DBS) and dried plasma spots (DPS). MS/MS detection was performed in positive ion ESI using MRM mode to monitor precursor→product ion transitions *m/z* 411.2→191.1 for RISP, *m/z* 427.2→207.1 for its metabolite RISP-9-OH. As per pharmacokinetic studies, the C_{max} of RISP and RISP-9-OH in DBS was 36.2 and 28.9 ng/mL; and in DPS was 38.2 and 30.2 ng/mL. Similarly, t_{max} of RISP and RISP-9-OH in DBS was 1.4 and 14.7 h; and in DPS was 1.54 and 15.4 h, respectively.

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SEPARATION SCIENCE PLUS / Volume 4, Issue 9 / p. 328-336

RESEARCH ARTICLE

Simple and rapid analysis of Linagliptin in dried blood spot using an ionic liquid based vortex-assisted dispersive liquid–liquid microextraction coupled with liquid chromatography–electrospray ionization–tandem mass spectrometry: Application to pharmacokinetic studies

Manikanta Swamy Arnipalli, Narendra Varma Nimmu, Ramachandra Bondigalla, Gangu Naidu Challa ✉

First published: 08 July 2021
https://doi.org/10.1002/sscp.202100008

Abstract

A simple, rapid, and convenient ionic liquid based vortex-assisted dispersive liquid–liquid micro extraction method for the determination of Linagliptin was developed. The main novelty of the present work deals with the analysis of Linagliptin in dried blood spot with significant advantages with regard to invasive sampling, volume of blood used (< 20 µL), storage and transport of biological materials and requirements for special biohazard arrangements. The extraction sample was assayed using liquid chromatography–tandem mass spectrometry using electropray ionization techniques. The effects of significant factors in extracting and disperser solvent, and salt contents were investigated. An efficient quantification method was developed for bioassay of Linagliptin using Sitagliptin as internal standard through Inertsil ODS-3V (250 mm × 4.6 mm, 5 µm) column using 10 mM ammonium acetate buffer (pH adjusted to 4.6 with 0.1% Trifluoroacetic acid): Acetonitrile [10:90 (v/v)] as mobile phase. Tandem mass spectrometry detection was performed to monitor precursor → product ion transitions *m/z* 473.3 → 364.2 for Linagliptin, *m/z* 408.2 → 235.1 for internal standard. The assay was linear from 6 to 1500 ng/mL and the mean recoveries are more than 90%. The peak dried blood spot concentration (C_{max}) after 1.5 h was determined to be 1391.2 ng/ml for Linagliptin.

CONFLICT OF INTEREST

The authors have declared no conflict of interest.

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1 Blech S, Ludwig-Schwellinger E, Gräfe-Mody EU, Withopf B, Wagner K. The metabolism and disposition of the oral dipeptidyl peptidase-4 inhibitor, linagliptin in humans. *Drug Metab Dispos*. 2010; **38**: 667–78.

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SEPARATION SCIENCE PLUS / Volume 4, Issue 9

FRONT COVER | Free Access

Front Cover: Simple and rapid analysis of Linagliptin in dried blood spot using an ionic liquid based vortex-assisted dispersive liquid–liquid microextraction coupled with liquid chromatography–electrospray ionization–tandem mass spectrometry: Application to pharmacokinetic studies

Manikanta Swamy Arnipalli, Narendra Varma Nimmu, Ramachandra Bondigalla, Gangu Naidu Challa

First published: 03 September 2021
https://doi.org/10.1002/sscp.202170040

Graphical Abstract

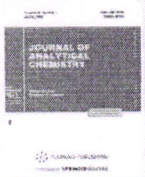
DOI: 10.1002/sscp.202100008

The cover picture shows a simple, rapid and convenient ionic liquid based vortex-assisted dispersive liquid-liquid microextraction method for the determination of Linagliptin. The extraction sample was assayed using liquid chromatography tandem-mass spectrometry using electrospray ionization. The effects of significant factors in extracting and disperser solvent, and salt contents were investigated. MS/MS detection was performed to monitor precursor → product ion transitions *m/z* 473.3 → 364.2 for Linagliptin, *m/z* 408.2 → 235.1 for internal standard. The assay was linear from 6–1500 ng/mL and the mean recoveries are more than 90%. The peak dried blood spot concentration (C_{max}) after 1.5 h was determined to be 1391.2 ng/mL for Linagliptin.

HPLC Bioassay of Elvitegravir using a Molecularly Imprinted Polymer Based Solid Phase Extraction in RAT Plasma: Application to Pharmacokinetic Studies

ARTICLES Published: 24 September 2021

Volume 76, pages 1172–1181, (2021) Cite this article



Nimmu Narendra Varma, Challa Gangu Naidu, Bondigalla Ramachandra & Arnipalli Manikanta Swamy

Abstract

A water-compatible molecularly imprinted polymer (**MIP**) was prepared for specific extraction of HIV-1 integrase inhibitor elvitegravir (**EVG**). It was prepared by a non-covalent free radical polymerization process using methacrylic acid as a monomer and elvitegravir as a template molecule. The MIP based solid phase extraction (**MIP-SPE**) cartridge was constructed for specific extraction of EVG from rat plasma samples. The effect of porogenic solvents, cross linker, pH and monomer to template ratio were studied. The developed HPLC method was validated as per ICH guidelines. The recovery of EVG using MIP-SPE technique was 98%. The LOD and LOQ of EVG were 0.01 and 0.05 µg/mL, respectively. The established method may not only be used to determine EVG but also to study the pharmacokinetics in rat plasma samples.

SEPARATION SCIENCE PLUS / Volume 6, Issue 4 / 220119

REVIEW ARTICLE

Characteristics, properties, and analytical and bio-analytical methods of enzalutamide: A review

Ramachandra Bondigalla, Gangu Naidu Challa, Srinivasa Rao Yarraguntla, Raju Bandu, Subba Reddy Alla

First published: 07 February 2023
https://doi.org/10.1002/sscp.202200119

Abstract

Enzalutamide is a potent second-generation androgen receptor inhibitor that is used to treat metastatic castration-resistant prostate cancer. It was developed by Medivation and Astellas and was approved by the Food and Drug Administration in 2012 under the brand name Xtandi. Enzalutamide has three major anticancer mechanisms, it inhibits the binding of androgens to the ligand-binding domain of androgen receptors; inhibits nuclear translocation of androgen receptors inhibits binding of androgen receptors to deoxyribonucleic acid. It demonstrates reduced expression of androgen receptor-dependent genes, decreased prostate cancer cell proliferation, and induction of cancer cell death and tumor regression. As a result, patients with metastatic castration-resistant prostate cancer have a higher chance of survival. Enzalutamide is very important in anticancer therapy, hence, it's necessary to compile all the analytical and bio-analytica methods to monitor the bioequivalence, bioavailability, and therapeutic monitoring of a drug substance during the course of patient follow-ups. Thus, this study presents a comprehensive review of the literature on characteristics, properties, and analytical and bio-analytical methods in various matrices, including formulations, biological fluids, and drug delivery systems.

CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

Open Research

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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FABRICATION OF SODIUM ALGINATE/GUM GHATTI IPN MICROBEADS INTERCALA KAOLIN NANO CLAY FOR CONTROLLED RELEASE OF CURCUMIN

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Keywords: Gum Ghatti, Kaolin, Sodium alginate, Curcumin, Microbeads

ABSTRACT

Objective: The objective of this study is to fabricate sodium alginate (SA)/gum ghatti (GG) microbeads intercalated with Kaolin (KA) nano clay for the sustained release of curcumin (CUR).

Methods: The microbeads were prepared by a simple ionotropic gelation technique. The developed beads were characterized by fourier transform infrared spectroscopy (FTIR), differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), X-ray diffraction (X-RD), and scanning electron microscopy (SEM). Swelling studies and *in vitro* release studies were investigated under both pH 7.4 and pH 1.2 at 37 °C.

Results: The developed microbeads were characterized by FTIR, which confirms the interaction between CUR, polymeric matrix and KA. DSC and XRD analysis reveals that the CUR has molecularly dispersed in the polymer matrix. *In vitro* results illustrated that microbeads were influenced by the pH of test media, which might be suitable for intestinal drug delivery. The drug release mechanism was analyzed by fitting the release data into different kinetic equations and n values are obtained in the range of 0.609-0.640, suggesting that the developed microbeads showed the non-Fickian diffusion type drug release.

Conclusion: These results clearly illustrated that the developed KA intercalated polymeric microbeads are potential drug carriers for the controlled release of CUR.

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Experimental and Computational Study of Thiophene Based Calamitic Liquid Crystals

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ABSTRACT

The structurally analogous calamitic mesogens 4-((4-(decyloxy) phenoxy) carbonyl) phenyl thiophene-2-carboxylate [2TWC10] and 4-(Thiophen-3-yl) phenyl 4-dodecylbenzoate [S12] based on thiophene were synthesized and structures of the molecules were confirmed by spectroscopic techniques. Among the two molecules, only 2TWC10 mesogen with alkoxy terminal exhibited a typical threaded structure indicating a homeotropic nematic phase under hot stage-polarizing optical microscopy (HOPM). Further, it is supported by differential scanning calorimetry (DSC). Remarkably, alkyl terminal S12 mesogen is not showing liquid crystalline properties. This is because S12 has alkyl group as the terminal group instead of alkoxy group which was used generally, resulting in bent shape to the molecule which reduced aspect ratio which is essential for liquid crystalline property. UV-Visible absorption maxima because of π-π* transitions in these mesogens were found at 280-300nm in chloroform solution. The DFT study shows that the alkoxy terminal in 2TW10 is contributing to polarity of the molecule but in S12 there is no contribution from terminal chain because it is non polar group. The DFT study also shows that 2TWC10 is more reactive and less stable than S12 molecule.

Keywords: Nematic phase, Photo-physical, Thiophene, DFT Study, Calamitic.

INTRODUCTION

Heterocyclic thiophene based liquid-crystalline materials found potential use in optical information storage, photovoltaic cells, organic thin-film transistors, spatial light-modulation, fast switching ferroelectric materials, in optical signal processing, fluorescent probes, chemosensors and so on[1-3]. The extent of use of liquid crystalline molecules in various fields depends on molecular

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Determination of Uric Acid Using TiO_2 Nanoparticles Modified Glassy Carbon Electrode

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Abstract: In the present study, the electrochemical behavior of uric acid (UA) was evaluated using a titania nanoparticle (TiO_2) coated glassy carbon electrode (GCE). TiO_2 nanoparticles are synthesized and characterized using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Analysis (EDX) techniques. The electrochemical behavior of uric acid on both bare GCE and Titania coated GCE electrodes were studied through differential pulse voltammetry. Titania-coated GCE showed a higher current at the lower potential for the oxidation of uric acid when compared to bare GCE. The sensor's improved electrocatalytic activity was observed to detect uric acid in a 0.1 M phosphate buffer saline (PBS) solution at pH 7.0. A good linear relationship was observed between electrical response and the concentration of uric acid in the range of 1 to 9 µM. Under optimized experimental conditions, the limit of detection (LOD) was found as 0.764 µM. The sensor has expressed considerable sensitivity towards UA detection without interference and is successfully used to determine UA in human urine samples.

Keywords: uric acid; electrocatalytic activity; TiO_2 nanoparticles; electrochemical sensor.

1. Introduction

Uric acid (UA) is an important nitrogen-containing compound present in both animals and plants. It comprises 2,6,8- trihydroxy purine, which exhibits keto-enol tautomerism and converts to the corresponding urate. The presence of more uric acid and urate leads to accumulation as calculi in the joints, and connective tissues cause arthritis and rheumatic pain, and excess deposition of uric acid and urate causes kidney failure. Uric acid is a biomolecule in urine and blood and is generated by breaking the purine in the body's metabolic process [1]. The normal level of uric acid in the blood is 0.12 mM – 0.45 mM, and in urine is 2 mM [2]. Increased uric acid levels in body fluids can cause diseases like toxemia of pregnancy, hyperuricemia, etc. [3-4]. Several studies suggested that the unusual UA level in blood serum is the risk factor, can cause cardiovascular disease, and influence the circulatory system [5]. In this context, UA sensing with commercial monitoring, anticipated sensitivity, and accuracy in human body fluids is crucial to disease sense. Different techniques, including HPLC, fluorescence, calorimetry, and direct electrochemical detection, have been developed to quantify uric acid in biological fluids. The electrochemical technique is widely used and has

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Short communication

Ethylene glycol-assisted synthesis of reduced graphene oxide-supported bimetallic Pt-Co nanoparticles for the ultra-sensitive detection of tert-butyl hydroquinone

B. Rajeswari [a], B. Sravani [b], Michael Cheffena [c], Ramavath Janraj Naik [d], Y. Veera Manohara Reddy [c d], G. Madhavi [f], K.V.N. Suresh Reddy [a], Myung Jong Kim [e]

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Highlights

- Facile synthesis of bimetallic Pt-Co/RGO nanocomposite.
- Pt-Co/RGO nanocomposite-based sensing platform was successfully constructed by this method.
- Fabricated sensor for precise determination of TBHQ in soybean, sunflower, and corn oils samples.

Abstract

In this study, we developed reduced graphene oxide-supported Pt-Co nanoparticles (Pt-Co/RGO) utilizing a straightforward one-step hydrothermal method and ethylene glycol as a shape-directing agent. We systematically examined how various experimental settings affected the morphology and structure of the Pt-Co/RGO nanohybrid material. Compared to Pt/RGO as prepared, Pt-Co/RGO has better catalytic activity. The electrochemical performance of the screen-printed electrodes (SPE) was evaluated through cyclic voltammetry (CV) and differential pulse voltammetry (DPV) measurements. The results show that, under ideal circumstances, the developed sensor can readily employ CV and DPV to detect tert-butyl hydroquinone (TBHQ). The designed sensor has a linear response from 5 nM to 100 nM with a detection limit (LOD) and quantification limit (LOQ) of 0.09 nM and 0.27 nM, respectively. The proposed sensor demonstrated its potential analytical performance by measuring TBHQ in samples spiked with soybean, sunflower, and corn oils, yielding good recoveries without sample pre-treatment.

Graphical abstract

FINANCIAL INCLUSION IN RURAL INDIA: THE ROLE OF MICROFINANCE

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ABSTRACT

Financial inclusion has been a key priority for India. Since finance serves as a catalyst for economic development, the relevance of financial inclusion stretches beyond the realm of finance to socioeconomic development. This paper main objective is to examine the relationship between financial inclusion and rural development through micro finance in India and to analyze the performance of micro finance- SHGs Bank Linkage Programme. In India, micro-Finance scene is dominated by Self Help Groups (SHGs) - Banks linkage Programme, aimed at providing a cost effective mechanism for providing financial services to the unreached poor. The focus of this sector must be on Digital Microfinance. Keeping in view the need to increase transparency, address customer-centric issues and safeguard the interests of low-income customers, microfinance lenders must put the interests of their clients first and implement the Code for Responsible Lending and the Code of Conduct in both letter and spirit. Redressing consumer complaints quickly and effectively it should be on top of the agenda for MFIs and the Self- Regulatory Organizations (SROs). It is concluded that the Financial Inclusion plays a vital role in providing economic opportunity and improves the living standard for inclusive growth. Micro-Finance is emerging as a powerful instrument for poverty alleviation in the new economy.

Keywords: *Financial Inclusion, Self-Help Groups (SHGs), Bank Linkage Programme and Self-Regulatory Organizations (SROs).*

INTRODUCTION

Financial inclusion has been a key priority for India. Since finance serves as a catalyst for economic development, the relevance of financial inclusion stretches beyond the realm of finance to socioeconomic development. Its benefits, thus, do not remain limited to the beneficiaries alone but are economy wide. Since its inclusion as a policy objective by the Reserve Bank of India (RBI) in 2005, numerous policy initiatives have been taken both by the RBI, and the Central and State