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Evaluation of the use of solvent impregnated resins in the analysis of salbutamol in human urine followed by capillary electrophoresis

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ABSTRACT

In this work, a method for sorption and concentration of salbutamol in urine combined to capillary electrophoresis with UV detection was performed. The procedure is based on the sorption of salbutamol on solvent-impregnated resins that is prepared by an impregnation technique using Aliquat 336 as extractant and XAD-4 resin as the base polymer. Batch studies showed an efficient sorption/desorption results when the salbutamol solution contains NaOH 0.05 mol L^{-1} and the eluent is 0.5 mol L^{-1} NaCl. Linearity was obtained in the range of 1000–10,000 ng mL^{-1} of salbutamol. The limit of quantification was 999 ng mL^{-1}. The solvent-impregnated resin was used for 10 cycles without a significant loss of the salbutamol quantification capacity. The method was applied to analyze salbutamol in urine samples at levels useful for international health organizations. Although most reports of solvent impregnated resins are related to the extraction of metal ions and only a few organic compounds, the proposed methodology demonstrate that solvent impregnated resins allows the isolation and concentration of salbutamol from complex samples such as urine. Since no SB is recovered using resin without Aliquat 336, this work shows an advantage of SIR over simple adsorption processes.

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1. Introduction

In recent years, solvent-impregnated resins (SIRs) have been considered good adsorbent materials since they are capable to sorb selectively a wide variety of compounds such as ion metals and organic compounds. SIRs can be considered as liquid extracting agents dispersed homogeneously in a solid polymeric medium. Because of an extractant agent is involved, it is possible to adapt the conventional extraction processes to a SIRs technique.

Some requirements involved on preparation of SIRs are: a liquid complexing agent that allows a specific or selective extraction, a solid polymeric support which contains the extractant agent, and a convenient and successful process of impregnation [1]. Impregnation process use a porous resin, such as polystyrene-divinylbenzene, immersed into a volatile organic solvent containing an extracting agent that should be liquid at the working temperature. The extracting phase exhibits a strong affinity to the polymeric matrix and the analyte, even after the subsequent evaporation of the organic solvent [2–3].

Some advantages of the use of SIRs are the ease of phase separation due to the elimination of problems dealing with the formation of stable emulsions, the possibility of designing and using a dynamic mode operation in columns with a continuous separation process, and the small amount of extracting agent and solvents used during the entire process [4].

As previously stated [5], SIRs have been successfully applied primarily to the sorption of metal ions and for a few organic compounds. However, in the case of drugs, the use of these resins has been limited to spyramicin [6–7]. For this reason, this paper aims at the application of SIRs as technological alternatives for the extraction and concentration of drugs such as salbutamol.

Salbutamol, or albuterol (SB) (2-(*tert*-butylamino)-1-(4-hydroxy-3-(hydroxymethyl)phenyl)ethanol), is an agonist typically used in the treatment of bronchial asthma due to its relaxing effect on smooth muscles and the subsequent relieving of airway obstructions [8]. In spite of its advantages, the use of salbutamol at higher dosage levels can cause tachyarrhythmias under conditions of hypoxia and hypokalemia, produce ischemic heart disease and cardiac failure, and cause an increased risk of cardiovascular death [9].

Also, due to lipolytic and anabolic effects, salbutamol has been illegally used as a growth promoter in the meat producing industry and as a doping agent in sports [10] where its presence in urine above 1000 ng mL^{-1} makes it suspect for a non-therapeutic use [11].

In most cases, the quantification of SB in urine requires the pretreatment of the sample by liquid-liquid extraction (LLE) [12] or solid phase extraction (SPE) with different types of no-polar cartridges (silica gel

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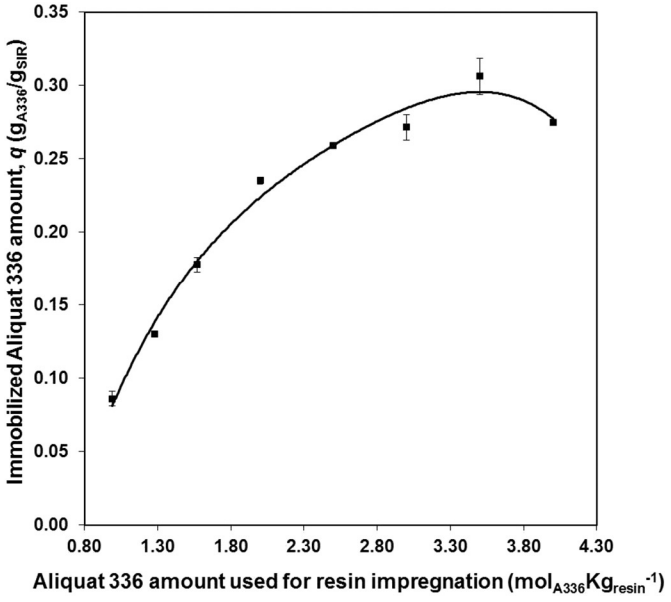

Fig. 1. Variation of the resin impregnation degree regarding the amount of extractant used to immobilize in the resin.

with octadecyl or carboxylic acid, SCX Extract-Clean, ASPEC, DECs, and polymeric Bond Elut PLEXATM SPE cartridge) [13–17]. Although these techniques are very popular for the separation and concentration of inorganic and organic substances, they have some disadvantages like the large consumption of solvents and the occasional clogging of the cartridges during the handling of real samples [18]. Another disadvantage of the common methods for the SB analysis is that it involves the use of techniques like HPLC-MS or GC–MS, costly and complicated in their operations [19–21].

As a result of the above mentioned, this paper shows a promising alternative for the sample pre-treatment and concentration of SB in urine based on the use of solvent-impregnated resins (SIRs) prepared by the impregnation of Aliquat 336 onto XAD-4 polymeric support. Several factors that have influence on the SB sorption and desorption, such as: amount of extractant in the resin, and composition of feed and stripping solutions, are evaluated and discussed.

2. Materials and methods

2.1. Materials

Chemicals like Aliquat 336 (A336) (Trioctylmethylammonium chloride), *n*-hexane, acetone, and sodium hydroxide were purchased from Aldrich. J.T. Baker supplied the other reagents. Water with resistivity ≥18.2 MΩ cm was obtained through a MilliQ Plus system (Millipore). A non-ionic crosslinked polymer, Amberlite XAD-4 resin, was from Rohm and Haas Company.

For a ready availability, salbutamol was obtained from pharmaceutical tablets acquired from a local drug store (4 mg of the active ingredient), and were used to prepare test and stock solutions. Concentrations of SB in stock solution were determined by interpolation in a calibration curve obtained by capillary electrophoresis technique using SB standards purchased from Aldrich. No differences in the number of signals and electromigration times were observed between the SB standard and the pharmaceutical product electropherograms.

2.2. Impregnation procedure

The impregnation procedure first involves washing the resin with HCl and acetone to remove potential impurities. Second, 0.5 g of Amberlite XAD-4 dry resin is immersed in 5 mL of A336 solution in hexane and stirred at room temperature. The A336/XAD-4 dry resin ratios ($mol_{A336}\ kg_{dry\ resin}^{-1}$) were varied in the range of 0.99 to 4. After 1 h, both phases are separated and the *n*-hexane is then slowly evaporated at 60 °C [22].

The amount of immobilized A336 extractant in the XAD-4/A336 SIR was represented as *q* and was calculated as (Eq. (1)) dividing the mass difference of the resin after (g_{SIR}) and before (g_{XAD-4}) the impregnation procedure, by the impregnated resin mass (g_{SIR}) [23]:

$$q = \frac{g_{SIR} - g_{XAD-4}}{g_{SIR}} \quad (1)$$

where g_{SIR} and g_{XAD-4} are the mass of resin after and before impregnation, respectively.

2.3. Sorption and desorption experiments in batch method

All experiments were performed at room temperature in triplicate. For batch sorption studies, an amount (0.25 to 1.0 g) of Aliquat 336 impregnated Amberlite XAD-4 resin (SIR) was added under agitation to a volume (25 to 200 mL) of feed solution (FS) containing NaOH (0 to 0.5 mol L^{-1}) and SB (5 mg L^{-1}). After a defined time, FS and SIR were separated, and the loaded SIR was mixed under agitation with a volume (2 to 5 mL) of a stripping solution (SS) containing NaCl (0.1 to 1.0 mol L^{-1}), achieving thereby the SB desorption in the SS. In both, sorption and desorption studies, agitation was set at 750 rpm. All batch experiments were performed during 2 h although equilibrium condition was reached at 7 h; the selected time did not affect the repeatability of the method. After every sorption and desorption study, the SB concentration was quantified in feed and stripping solutions by capillary electrophoresis.

The percentage of SB sorbed (extracted) to SIR ($\%S_{SB}$) was calculated with the following equation, where $SB_{FS,initial}$ is the concentration of salbutamol in feed solution before the sorption process (5 mg L^{-1}),

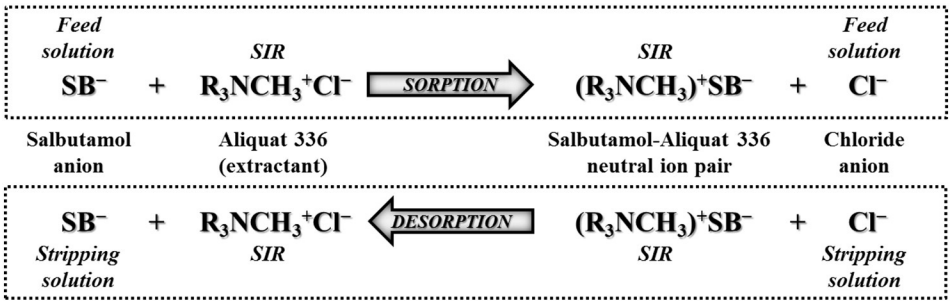

Fig. 2. Mechanism of sorption and desorption of SB with SIR containing Aliquat 336 as extractant.

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