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Development and validation of an analytical method for quantitative determination of carboxylic acids in air samplers

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Abstract

The aim of this article was to realize a method for the determination and separation of formic and acetic acids in air samplers. For this purpose, an analytical ion chromatography method was developed, optimized under various measurement conditions and then validated. Selectivity, linearity, limit of detection, limit of quantification, precision and accuracy of the method were determined for the validation process. The resulted method can separate and determine the interested compounds, is fast, simple to reproduce and involves low reagent's costs.

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1. Introduction

The indoor environmental quality is very important because it has an direct influence on the occupants comfort and productivity [1, 2]. One of the most harming indoor pollutants are VOC and formaldehydes. The volatile organic compounds (VOCs) and formaldehyde are organic chemicals that have a high vapor pressure at ordinary room temperature. These substances are used to manufacture and maintain building materials, furniture, maintenance products and personal care products [3].

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However, VOCs released from the wood products has been a long-studied problem. It is important to control and decrease VOCs concentration used during the production stage.

Along with the wood products, paints, finishes and cleaning products are emitting VOCs, such as toluene, benzene, formaldehyde [4]. These substances have been indentified to be associated with asthma, nasopharyngeal cancer and multiple subjective health complaints [5]. The impact on the human health influenced international organism such as EU to set maximum values for the emissions of volatile organic compounds [4] and several air sampling strategies are proposed.

Based on the human perception the response regarding the small variation of temperature or relative humidity is not the same. Thus occupants can report a "dry sensation" or irritation of respiration system due to a polluted environment accusing air humidity variations [5]. VOC can be identified by occupants based on odor but in [6] there is presented that the occupants are not able to identify an odor source, because they are living in an environment with odors from non-natural sources. Therefore they are not able to identify the sources that have an impact on their health. Any questionnaires that are used to identify the sources might be involuntary partially completed.

The formaldehyde is a natural metabolic product of the human body, exposure to high-dose increases the risk of acute poisoning. The International Agency for Research on Cancer in 2006 stated that long exposure can lead to chronic toxicity and even cancer. Therefore it is needed to detect and limit the concentration of formaldehyde at source (product emission) and remove if the concentration is high. Formaldehyde levels between 0.1 and 0.5 ppm (0.12–0.6 mg/m3) are detectable by human senses, between 0.5 and 1.0 ppm (0.6–1.2 mg/m3) can cause eye irritation, and above 1.0 ppm (1.23 mg/m3) can irritate the nose and throat [7].

The scope of this article is to present an method to evaluate the indoor air quality based on the formaldehyde presence in the air. The assessment is based on the concentration of formic and acetic acids. These acids are the results of the formaldehyde natural oxidation process.

2. Materials and methods

The analytical method develop for the separation and determinations of the two carboxylic acids (formic and acetic) was realized on a Dionex ICS-5000+ Ion chromatograph model, with integrated eluent generator, a conductivity detector and anion suppressor model 500 ORSA Dionex 2mm.

To separate the acids, an IonPac AS-18 column chromatography coupled with a precolumns recommended by the manufacturer to determine anions and low molecular weight organic acids were used.

Elution mode was isocratic, and consisted of 10 mM of KOH as eluent. The thermostatic column was set at 20 °C and the injection volume was 5µl; the total running time of the method was 10 minutes.

The calibration curves were realized using external standard method. For this porpoise, analytical standards were purchase from LGC Standards, and consisted in 1000 mg/mL acetate and 1000 mg/mL formate.

3. Results and discussions

The results presents the method validation. The method's parameters determine for the validation were: selectivity, linearity, limit of detection, limit of quantification, precision and accuracy.

The chromatographic columns plays an important role in the separation of chemical compounds. For this purpose, an IonPac AS-18 was chosen. This column have been described as being suitable for determination of low molecular weight organic acids like formic and acetic acids. IonPac AS Columns had been characterized in literature as having good results in separating formic and acetic acids and being able to determine this compounds in air samplers[8].

Along with the chromatographic column, the eluent concentration can make the difference between a good or an incomplete separation of the compound's peaks. We started by testing different eluent concentrations on KOH, from 23mM to 2mM.

In Figure 1is presented the two carboxylic acids with eluent concentration of 23mM KOH. In this case, the resolution between the peaks is lower than 2 and, as can be seen, the peaks are not completely separated at the base.