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An in-needle extraction technique in determination of organic compounds released from dental tissue conditioners incubated in artificial saliva

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ABSTRACT

The use of an in-needle technique for direct isolation of analytes from real liquid samples is a new proposal. The in-needle technique has been relatively seldom used for direct sampling of liquid matrix through the needle. In this work the in-needle technique has been applied for the determination of compounds evolved to artificial saliva from dental prosthetic materials. It has been shown that results from the experiment with in-needle device were at least comparable with those obtained with using well known solid phase extraction (SPE). It is worth to mention that in-needle extraction offers some advantages: lower consumption of solvent, shorter step-preparation time and reduced costs. The compounds released from prosthetic materials may affect the stability of tissue conditioners and limit their long-term use in the oral cavity. Examined soft dental materials have been found to be stable as minor amount of various species have been emitted from them. Results of the stability tests of soft dental materials with the use of in-needle device on sample preparation step enable their quick evaluation and estimations of their quality.

1. Introduction

Sample preparation is an important analytical step. Isolation of components and their concentration to a level enabling the quantitative determination is a major problem in most of modern analytical procedures. Conventional sample preparation methods most often require large amount of organic solvents, are relatively complicated and time-consuming. Two sample preparation techniques were used in this work: solid phase extraction (SPE) and an in-needle technique. By proper designing the in-needle system it was possible to avoid the main drawbacks of SPE [1] and SPME [2], such as labor expense and the need of careful handling of expensive.

Solid-phase extraction was used successfully and widely for the preparation of liquid samples. SPE was applied in many studies because, in comparison with other conventional methods, it has a lot of advantages. However, some limitations and drawbacks of SPE caused that more effective methods of sample preparation are developed continuously. In-needle extraction has many advantages. Therefore, it is desired to apply this technique to the direct liquid sample preparation. First of all, in-needle device is much cheaper. Moreover, the amount of solvent can be reduced to less than 1 mL and an in-needle extraction device can be used on-site. Its mobility might be treated as another advantage.

The needle trap device (NTD) was described in the literature very thoroughly, see e.g. [3–11] but over the years the in-needle technique was used to prepare gaseous samples. Isolation of analytes from water samples was most often combined with head-space (HS) or purge-and trap- (P&T) techniques, probably due to high flow resistance produced by a sorbent layer. Moreover, only a portion of analytes was passed through the sorbent bed, leading to problems in quantitative analysis and a possible loss of the sample components.

The novelty of the proposed in-needle device lies in the reduction of solvent consumption, cost reduction and possibility of the prediction of device efficiency basing on its macroscopic properties.

1.1. Direct analysis of the liquid sample

The resistance resulting from the flow through porous material causes considerable prolongation of the extraction process. The efficacy of the in-needle extraction device may be increased by using smaller sorbent particles of packing. This will be, probably, related to better recovery of the analyte. However, smaller size of particles restricts the flow rate and limits the sampling speed.

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Therefore, parameters affecting the efficiency of extraction should be examined and optimized.

Saito et al. prepared the extraction device, with a bundle of the polymer-coated filaments as the sorbent material packed into a needle. The extraction was made by pumping the aqueous sample solution into the needle extraction device. The sample flow rate during the extraction was optimized and it was found that the flow rate should be about 16 μL/min, which is equivalent to 1 mL/h [12].

In 2012 Pietrzyńska et al. [13] proposed a direct use of the in-needle technique for the preparation of liquid samples by passing water samples directly through the needle filled with a sorbent. The effectiveness of the NTD system was studied based on experimental data and chemometric evaluation.

The quantitative criteria for selection of parameters of the NTD system were derived by Kaczmarek et al. [14]. The conditions were formulated for the force exerted on syringe, the volume of tested solution, for the time of test and contact time of solution with the sorbent. The last two conditions allowed to establish limits for combinations of fundamental geometrical and macroscopic structural characteristics of the system.

1.2. Prosthetic materials

Tissue conditioners belong to a group of prosthetic materials that are used during the treatment of patients wearing removable partial or complete dentures. They release to the oral cavity compounds that may, when occurring in certain amounts, have a negative influence on human health. The factors accelerating their release may include liquids such as saliva or disinfectants, which, in contact with the prosthetic material, can penetrate the polymer network. Amount of released potentially dangerous compounds should be determined to obtain accurate information regarding the harmful effects of prosthetic materials on the human body.

In dental prosthetics, tissue conditioners are used for short-term relining of removable dentures. With time, the mucosal surface of a relined denture changes its shape adapting to the configuration of the soft tissues of the prosthetic base, which can lead to faster healing of the soft tissues, for example after a surgery. Tissue conditioners may also be applied in taking functional impressions. Patient, using a relined denture for some time, shapes its surface under physiological conditions. Such materials exist as powders (e.g., ethyl polymethacrylate) and liquids (monomers, plasticizers, and alcohols). After mixing both ingredients, bridges between the polymer particles are made and a three-dimensional network is created. In the case of polymers of low molecular mass, the cross-linking proceeds to a small extent, and the resulting substance takes the form of gel. Alcohol accelerates the process. The elution of alcohol and plasticizers leads to a quick deterioration of the adhesiveness and elasticity of tissue conditioners. Over time they become as hard as the denture corpus, which may lead to irritation and damage of the soft tissues of the base. In certain concentration plasticizers may show hormonal activity towards the human body tissues, acting as xenoestrogens. They demonstrate an ability to interact with the endocrine system and to modulate its activity in a way characteristic of estrogens. Therefore, it seemed advisable to conduct laboratory studies in order to evaluate qualitatively and quantitatively the chemical compounds that are eluted from tissue conditioners.

1.3. The aim of this study

The aim of this study was to determine the possibility of using in-needle device for direct liquid samples preparation and to identify as well as quantitatively determine the compounds released from tissue conditioners incubated in artificial saliva.

2. Materials and methods

2.1. Materials

The ageing of two commonly used soft lining materials, Softone (Bosworth Comp.) and Visco-Gel (Dentsply DeTrey), was examined (Table 1).

The methanol p.a., and dichloromethane p.a. were obtained from Polskie Odczynniki Chemiczne S.A. (Gliwice, Poland). The water was purified by the membrane technique using a RO5max system for water deionization (Bichmitte, Poland). The components for SAGF, i.e. Gal-Fovet artificial saliva [15] listed in Table 2 were obtained from Chempur (Poland).

The stainless steel needles with an internal diameter of 2.7 mm and the 10 mL gas-tight syringe were products of Danlab, Poland. The styrene–divinylbenzene copolymer (SDB-1) (J.T. Baker, Deventer, Holland) was supplied by Witko, Łódź, Poland. The SPE columns with the SDB sorbent (200 mg) were manufactured by J.T. Baker, Deventer, Holland.

2.2. Methods

2.2.1. Needle preparation

The filling needle process consists of 3 stages. First stage concerns the introduction of the first supporting layer into needle. Then, needle was filled with sorbent material with the dry pack

Table 2
The components of SAGF (pH 6.8), buffer 0.1 N NaOH or 0.1 N HCl.

Component	Concentration (mg/L)
NaCl	125.6
$NaHCO_3$	630.8
KCl	963.9
KH_2PO_4	654.5
KSCN	189.2
$CO(NH_2)_2$	200
$CaCl_2 \cdot 2H_2O$	227.8
NA_2SO_4	763.2
NH_4Cl	178

Table 1
Prosthetic materials used as tissue conditioners.

Marketing name	Producer	Components (%, m/m)			
		Powder		Liquid	
Softone	Harry J. Bosworth Company (USA)	Poly (ethyl methacrylate)	N/A	Acetyl phthalate	N/A
				Ethanol	N/A
Visco-Gel	Dentsply DeTrey (Germany)	Poly (ethyl methacrylate)	50–100%	Paraffin oils	50–100%
				Ethanol	2.5–10%