



# Synthesis and characterization of composite based on cellulose acetate and hydroxyapatite application to the absorption of harmful substances

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## ABSTRACT

The aim of this work is to develop composite materials with hydroxyapatite (HAp) mineral and organic matrix such as cellulosic polymers. We use cellulose acetate with different percentages, and then inorganic–organic films were fabricated by evaporation of solvent. The composite films were characterized using emission scanning electron microscopy (FEG-SEM), thermo-gravimetric analysis (TGA) and Fourier transform infra-red (FT-IR) spectra. Test results show that these films are uniform and have good ductility. A strong interaction existed between HAp and cellulosic polymers, and the method allows the production of very fine particles size of about 92 nm. We have developed a new chromatographic method for the quantification of bisphenol A (BPA) in samples of baby food. The result of this study demonstrates how to use this type of composite materials to remove pollutants.

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## 1. Introduction

Recently, composite materials based on calcium phosphate have attracted much attention (Azzaoui, Berrabah, et al., 2013; Azzaoui, Lamhamdi, et al., 2013; Needham, Calafat, & Barr, 2007). Hydroxyapatite is often used as a bone implant material; however, it is used to develop many composites for advanced technologies. Many studies that use bio monitoring used data on food contamination bisphenol A and the environment (Angerer, Ewers, & Wilhelm, 2007; Boogaard, 2007; Calafat, Ye, Silva, Kuklennyik, & Needham, 2006; Needham et al., 2007; Pirkle, Osterloh, Needham, & Sampson, 2005; Yang, Park, & Lee, 2006). This information served as the basis for estimating indirect human exposure to bisphenol A and its potential toxicity. Cellulose acetate is considered

one of the essential esters of cellulose. According to the methods used, it can be widely used in different kinds of purposes; such as film formation and membranes. The toxicity of bisphenol A has been extensively studied covering a large range of doses; the study showed adverse effects at doses of 50 mg/kg body weight/day (EU, 2003; Goodman et al., 2006; Gray et al., 2004).

The technology employed for the extraction of BPA from aqueous samples includes solid phase micro-extraction (SPME) (Kasuga et al., 2003), liquid–liquid extraction (LLE) (Deng, Hao, & Wang, 2001) and solid-phase extraction (SPE) (Shafer & Simonian, 2002).

In this work, a novel analytical method based on enrichment and pretreatment of analytes in the water sample CA/HAp sorptive extraction and gas chromatography–mass spectrometry SIM mode have been developed for the rapid analysis of BPA in water. The obtained results demonstrated that CA/HAp/PEG 1000 combined with GC–MS is a simple, rapid and solvent-free method for the analysis of BPA in water.

The aim of this work was then to choose the best stationary phase (TFME) and best chromatographic method for the

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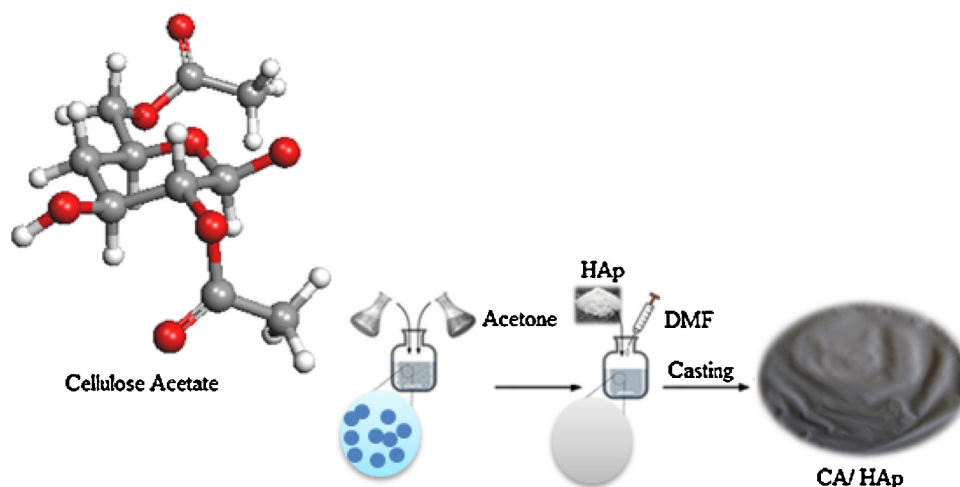


Fig. 1. Schematic representation of synthesis route of composite.

quantification of BPA in samples of baby food matrices, which could be used for routine controls.

## 2. Experimental and methods

### 2.1. Materials

Hydroxyapatite (HAp) was synthesized in our laboratory. The Cellulose Acetate (CA) (99%), Calcium Nitrate  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  (99%), Ammonium Hydrogen Phosphate  $(\text{NH}_4)_2 \cdot \text{HPO}_4$  (99%), DMF (Dimethylformamide) and Acetone were purchased from Aldrich. High purity distilled water was used throughout the whole experiment.

### 2.2. Synthesis of thin film; CA/HAp/PEG1000

The starting substances used in the production of hydroxyapatite were calcium nitrate tetra hydrate and di-ammonium hydrogen phosphate. HAp was produced by following a modified wet chemical method (Azzaoui, Berrabah, et al., 2013; Azzaoui, Lamhamdi, et al., 2013). At 25 °C, 11.76 g of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  was first dissolved in a 100 ml volume of water. A solution of 4.06 g  $(\text{NH}_4)_2 \cdot \text{HPO}_4$  was dissolved in 100 ml volume of water and then added to the  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  solution over a period of 30 min. The amount of reagents in the solution was calculated to obtain a Ca/P molar ratio value equals 1.67, corresponding to a stoichiometric HAp. About of the polyethylene glycol was added along with the mixture. The pH of the slurry was measured digitally during the precipitation reaction, reaching a final value of pH 10.5.

Cellulose Acetate (CA) polymer were dissolved in acetone at room temperature to form solution A. Solution B was hydroxyapatite (HAp) dispersed in DMF at room temperature and added to solution A. The solution turned to opaque milky white after about half an hour. Thin films were obtained during the procedure with careful temperature control to 50 °C at the temperature rate of 2 °C min<sup>-1</sup> and maintained for 2 h, and then cleaned in absolute ethanol to remove any possible impurities in the film (Fig. 1).

### 2.3. Preparation of standards and sample solution

The standard stock solution was prepared by accurately weighing 100 mg of BPA into 1 L (S0) volumetric flask and dissolved in distilled water. The stock solution was diluted with distilled water to obtain solutions S1 and S2, 10 and 1 mg/L, respectively.

Prior to use thin powders were washed with the deionised water, absolute ethanol and dried in the oven at 120 °C for 1 h in order to remove impurities and any possible residual analytes (interferences). Extraction and desorption were carried out as follows: thin powders were immersed in 50 mL of S2 in a glass vial under stirring (contact 20 min), after extraction, the thin powders were taken out from water, gently dried with a filter paper and put in an oven to dry at 40 °C for 40 min. finally, the thin s powders were directly placed in centrifuge tube (1.5 ml) to desorb and derive target analyte BPA with BSTFA 1% TMCS (200 µL) under sonication and heated at 70 °C for 30 min. Of the final 0.2 mL final derivative solution, 1 µL was directly injected into the Shimadzu GC/MS system for analysis. In each experiment, the analyte concentration was the same and each solution was analyzed in triplicate.

### 2.4. Conditions of BPA analysis

For the GC/MS system, a Shimadzu GCMS-QP2010 (Shimadzu, Japan) with GCMS solution 2.5 software was used. The Chromatographic conditions: GC analysis was performed on a Shimadzu QP 2010 system with a fused silica capillary column (30 m, 0.25 mm i.d. and film thickness of 0.25 mm with chemically bonded phase DB-5). The oven temperature was held at 60 °C for 1 min, programmed to rise at 10 °C min<sup>-1</sup> to 280 °C, and held for 5 min. A sample volume of 0.1–1 µL was injected in splitless mode (high pressure). The injector and interface temperature was kept at 250 °C. Helium was used as the carrier gas. Spectrometric conditions: mass spectrometric parameters: the electron impact ionization energy was 70 eV, the detector voltage 1.7 kV, the ion source temperature was 200 °C and the solvent delay time was 3 min. MS detector was used in multiple ion monitoring mode (ions characteristic of BPA in SIM mode were:  $m/z = 357$  and  $372$ ) (Azzaoui, Berrabah, et al., 2013; Azzaoui, Lamhamdi, et al., 2013).

### 2.5. Physical–chemical characterization

The prepared samples were characterized by making use of Fourier transform infrared spectroscopy (FTIR) using a Shimadzu FT-IR 300 series instrument (Shimadzu Scientific Instruments). FTIR spectra were acquired over the region 400–4000 cm<sup>-1</sup> in pellet form for 1 mg powder samples mixed with 200 mg spectroscopic grade (KBr). The structure of the samples were evaluated by X-ray diffraction (XRD) using a Rich Siefert 3000 diffractometer (Seifert,

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