

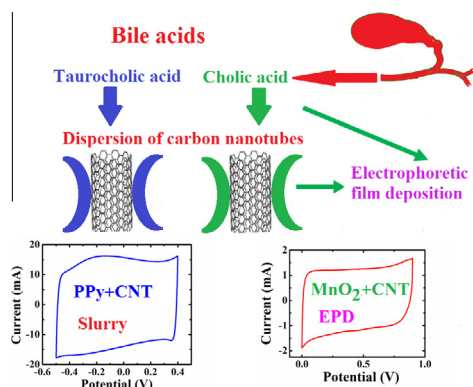
Colloidal methods for the fabrication of carbon nanotube–manganese dioxide and carbon nanotube–polypyrrole composites using bile acids



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GRAPHICAL ABSTRACT



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ABSTRACT

Nature inspired strategies have been developed for the colloidal processing of advanced composites for supercapacitor applications. New approach was based on the use of commercially available bile acid salts, such as sodium cholate (ChNa) and taurocholic acid sodium salt (TChNa). It was demonstrated that cholic acid (ChH) films can be obtained by electrophoretic deposition (EPD) from ChNa solutions. The analysis of deposition yield, quartz crystal microbalance and cyclic voltammetry data provided an insight into the anodic deposition mechanism. The outstanding suspension stability of multiwalled carbon nanotubes (MWCNT), achieved using bile acids as anionic dispersants, allowed the fabrication of MWCNT films by EPD. The use of ChNa for EPD offered advantages of binding and film forming properties of this material. Composite MnO_2 -MWCNT films, prepared using ChNa as a dispersant and film forming agent for EPD, showed promising capacitive behavior. In another colloidal strategy, TChNa was used as a dispersant for MWCNT for the fabrication of polypyrrole (PPy) coated MWCNT. The use of PPy coated MWCNT allowed the fabrication of electrodes with high active mass loading, high capacitance and excellent capacitance retention at high charge–discharge rates.

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1. Introduction

The development of colloidal nanotechnologies requires urgent breakthroughs in the methods of nanoparticle synthesis, dispersion and surface modification. The fundamental investigation of natural materials is an important source for new ideas in this area.

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Among the important examples are investigations, focused on applications of bile acids and their derivatives for the synthesis and dispersion of nanomaterials, which attracted significant interest of different scientific communities.

Bile acids, such as cholic acid and deoxycholic acid, were found to be efficient reducing [1] and chelating [2] agents for the synthesis of nanoparticles. Of special interest are gel-forming properties of bile acids, which were utilized [3] for the fabrication of hybrid Ag–Au nanoparticles with particle size of 1–2 nm and nanoparticles of ZnS and CdS with typical size of 3–10 nm. Deoxycholic acid has been utilized for the synthesis of CdSe nanocrystals with controlled nanodot or nanorod morphologies, which exhibited enhanced photoluminescence [3]. Deoxycholamide crystalline network [4] was used as a platform for the assembly of dye molecules, which showed high fluorescence quantum yields. Deoxycholate was found to be an efficient stabilizer of luminescent silicon nanocrystals [5].

Several investigations were focused on the application of bile acids and their derivatives for the development of photovoltaic devices [6–8]. These studies revealed the advantages of cholanic acids, compared to commercial surfactants, for the fabrication of dye-sensitized solar cells [6]. The use of cholanic acids allowed the fabrication of devices with enhanced photocurrent and voltage [6]. It was suggested that enhanced photovoltaic properties resulted from the positive shift [6] of the conduction band of TiO₂ induced by the adsorbed cholanic acid molecules and their 'spacer effect' on dye adsorption. The scanning tunnelling microscopy investigations [9] of adsorption of black dye and deoxycholic acid on TiO₂ showed that deoxycholic acid increased the number ratio of single dyes to aggregated dyes on the TiO₂ surface. The lower dye aggregation allowed reduced intermolecular electron transfer between the neighboring dyes and resulted in improved device efficiency [6,10].

Bile acids are powerful natural surfactants, which solubilize proteins, fatty acids, lipids, vitamins, monoglycerides and cholesterol [11]. The dissolving power of bile acids is related to their steroid molecular structure [12]. This structure is fundamentally different from the structure of conventional head-and-tail surfactants, which are composed of a hydrophobic tail and a polar head group. In contrast, bile acids have concave hydrophilic and convex hydrophobic surfaces [13]. Such surfaces play an important role in the interactions of bile acids with other molecules. Bile acids readily form mixed micelles with surfactants [14] and intermolecular compounds with various organic molecules [12]. It was found that

cholic and deoxycholic acids crystallized to form channels in which the second component of the intermolecular compound can be closely confined [12,15].

Many investigations were focused on the interactions of bile salts with single walled carbon nanotubes (SWCNT) and analysis of SWCNT dispersion in bile salts solutions [16–18]. It was found that cholates ions wrap around the SWCNT to form a ring. This is in contrast to conventional linear head-and-tail surfactants, which are usually oriented perpendicular to the SWCNT surface [16]. It was found that due to the unique structure and orientation of bile salts at the SWCNT surface, they allowed superior SWCNT dispersion, compared to the linear head-and-tail surfactants [16]. In another investigation [17], various commercial dispersants were compared and it was found that bile salts outperform all the investigated dispersants in the solubilization of SWCNT.

The use of bile acids opens new and unexplored routes to the fabrication of nanocomposites. The goal of this investigation was the application of bile acid salts for the fabrication of composites, containing multiwalled carbon nanotubes (MWCNT) for application in electrodes of electrochemical supercapacitors. Two different strategies were developed for the fabrication of thin film electrodes and bulk electrodes. We demonstrate that pure cholic acid films can be obtained from the solutions of sodium cholate by anodic electrophoretic deposition (EPD). The data analysis provides an insight into the influence of anionic ligands on the deposition mechanism. The results presented below indicate that cholate can be used as dispersing, charging, film forming and binding agents for the deposition of MWCNT. As an extension of these investigations we demonstrate the EPD of composite MnO₂–MWCNT films. In another strategy, taurocholic acid was used as a dispersing agent for colloidal fabrication of polypyrrole (PPy) coated MWCNT. Due to good dispersion of MWCNT, the composites, prepared by colloidal methods showed promising performance for application in electrodes of electrochemical supercapacitors.

2. Experimental procedures

2.1. Materials

Sodium cholate (ChNa), taurocholic acid sodium salt (TChNa), KMnO₄, pyrrole (Py), ammonium persulfate ((NH₄)₂S₂O₈) (APS), Tiron, (Aldrich), and MWCNT (Bayer) were used as starting materials. The MWCNT had average inner and outer diameters of about 4 and 13 nm, respectively, length of 1–2 μm and C-purity above 99%. Manganese dioxide nanoparticles with average particle size of 30 nm and Mn oxidation state of 3.6 were prepared by the reduction of aqueous KMnO₄ solutions with ethanol using a method described in a previous investigation [19]. PPy coated MWCNT

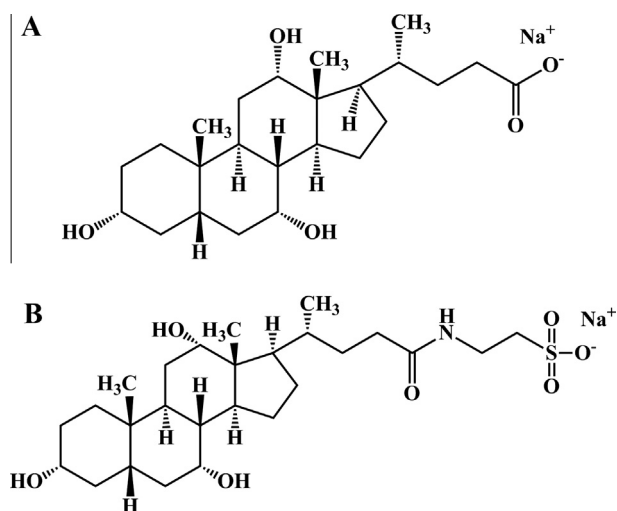


Fig. 1. Chemical structures of (A) ChNa (B) TChNa.

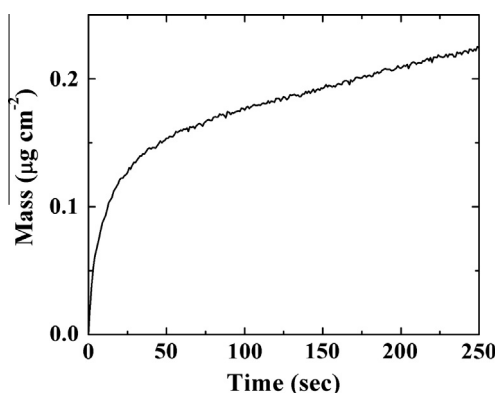


Fig. 2. Deposit mass versus time measured by QCM for 0.1 g L⁻¹ ChNa solution at a deposition voltage of 3 V.

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