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Film deposition mechanisms and properties of optically active chelating polymer and composites



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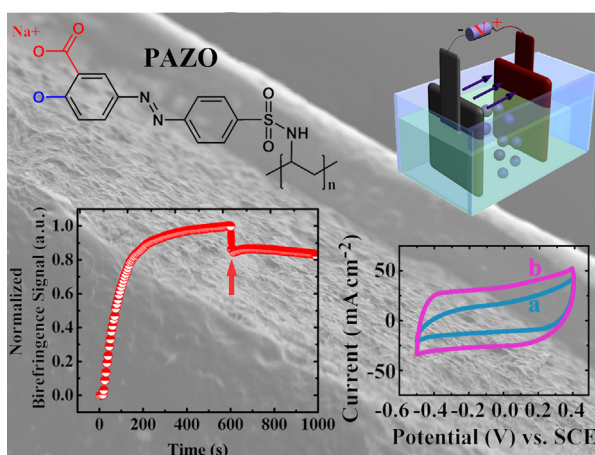
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HIGHLIGHTS

- Films of chelating azopolymer were deposited by electrophoretic deposition (EPD).
- The films exhibited photoinduced birefringence.
- The polymer showed strong adsorption on various organic and inorganic materials.
- The polymer was used as a dispersant for EPD of materials and composites.
- Optical and electrochemical properties of materials and composites were investigated.

GRAPHICAL ABSTRACT



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ABSTRACT

Poly[1-[4-(3-carboxy-4-hydroxyphenylazo)benzenesulfonamido]-1,2-ethanediyl, sodium salt] (PAZO) exhibits a number of unique physical properties. An electrophoretic deposition (EPD) method has been utilized for the deposition of PAZO films, which exhibited photoinduced birefringence. The advantages of EPD, deposition mechanism and kinetics of deposition are discussed. PAZO exhibits unique adsorption properties, which are related to its chemical structure. The salicylate ligands of PAZO monomers with strong chelating properties provide numerous adsorption sites for PAZO adsorption on inorganic materials. It was found that PAZO can function as a charging, dispersing and film forming agent for EPD of a wide array of materials, such as TiO₂ (anatase and rutile), micron size platelets and submicrometre particles of Al₂O₃, zirconia, MnO₂, MgAl₂O₄, aluminium hydroxide, SiC, Pd metal, carbon nanotubes and polypyrrole nanofibers. The feasibility of incorporation of other polymers and organic dyes into the PAZO films has been demonstrated. PAZO showed superior performance compared to other dispersing agents for the EPD method, overcoming their limitations. Another major finding of this study was the possibility of EPD of composites, using PAZO as a co-dispersant for different materials. Testing results showed that composites can be used for optical, energy storage, corrosion protection and other applications.

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1. Introduction

Poly[1-[4-(3-carboxy-4-hydroxyphenylazo)benzenesulfonamido]-1,2-ethanediyl, sodium salt] (PAZO) polyelectrolyte exhibits a unique set of properties, allowing for its diverse application in photonics, optoelectronics, memory devices and sensors as an advanced functional material [1–3]. Of particular interest are investigations of photo-induced birefringence in PAZO films [1], resulting from reversible *trans-cis-trans* photoisomerization of azo groups. A significant amount of research was focused on the analysis of optical storage and photochromic properties, surface-relief grating [4–7], azobenzene *trans-cis-trans* transitions and birefringence [4]. It was found that the transformation from the *trans* form to the *cis* form results in a change of the dipole moment of the PAZO molecules, which has been utilized in light sensitive biosensors [2]. Moreover, PAZO is an effective polymer film material for holographic surface patterning [8].

Many investigations were focused on the development of film fabrication methods. The layer-by-layer (LbL) adsorption method has been developed for the fabrication of films, containing anionic PAZO macromolecules and various cationic polyelectrolytes, such as poly (diallyldimethylammonium chloride) [2,9–11], poly(ethylenimine) [9,12], poly(allylamine hydrochloride) [11,13], poly(propylenimine) dendrimers [14] and ionenes [15]. The LbL method has been utilized for the fabrication of photo-tunable and photo-responsive polymeric microshells, covered by Au nanoparticles [16], and UV responsive microcapsules [11,17]. However, the electrostatic interaction of polyelectrolytes in LbL films has been observed to retard photo-isomerization and molecular rearrangement within the film, resulting in sluggish writing kinetics for photo-induced birefringence [5]. Moreover, the LbL method requires multiple and time consuming steps [18–20]. The need for faster processes has resulted in the development of modified, more temporally efficient LbL techniques [18]. Significant interest has also been focused on the development of other deposition methods. PAZO films have been prepared by casting, drop casting and spin coating methods, using PAZO solutions in organic solvents [1,4,8,21]. Composite films were prepared containing dyes [1,22,23], layered double hydroxides [24], enzymes [25], surfactants [26] and neutral polymers [27] in the PAZO matrix. Liquid crystal photoalignment was achieved in PAZO-liquid crystal composite films, which were subsequently used for the fabrication of thin film displays [28]. The synthesis of inorganic particles in the presence of PAZO showed that the morphology of the particles can be modified by *in-situ cis-trans* photoisomerization of the azobenzene groups [29]. The impressive progress achieved in application of PAZO and composite films has generated interest in the development of new film deposition methods and new composites.

Of particular interest are important structural features of PAZO, which the literature has not paid sufficient attention to. The structure of PAZO monomers includes a salicylate ligand (Fig. 1A(a)), containing COO⁻ and OH groups, bonded to the adjacent carbon atoms of the aromatic ring. Previous investigations of small molecules from the salicylic acid family showed their adsorption on inorganic particles, such as TiO₂ [30–35], iron oxides [36,37], Al₂O₃ [31,38,39], ZrO₂ [31], Ta₂O₅ [31], MnO₂ [40]. Adsorption mechanisms [31,32,35,40] have been suggested, such as bidentate chelating bonding or bidentate bridging bonding, which involved the salicylate ligands of the molecules and metal atoms on the particle surface. The adsorption of salicylic acid on TiO₂ resulted in improved optical and charge transfer properties [32]. Anionic molecules of salicylic acid, 5-sulfosalicylic acid 2,6-dihydroxybenzoic acid [33,40,41] were used for dispersion and EPD of MnO₂ and TiO₂. The adsorption of the salicylates on the particle surfaces was of critical importance for efficient dispersion and electrophoretic deposition (EPD) of inorganic materials. Building

on these results, other salicylates, such as 2-hydroxy-1-naphthoic acid, alizarin yellow, aurintricarboxylic acid and calconcarboxylic acid [42–46], were utilized for the dispersion and EPD of inorganic oxide particles. The use of aromatic molecules, containing salicylate ligands, allowed for their application as co-dispersants for inorganic particles and carbon nanotubes [46]. In this strategy the π - π interactions promoted dispersant adsorption on carbon nanotubes, whereas the complexation of the dispersants' salicylate ligands with metal atoms on the particle surface resulted in adsorption on the metal oxide nanoparticles. The method has been utilized for the fabrication of supercapacitor electrodes with improved capacitance and reduced impedance [46]. The use of aluminum and pamoic acid sodium salt [42] allowed for the EPD of carbon nanotubes, graphene, MnO₂ and TiO₂. However, so far this approach has had limited applications due to relatively weak interactions of single salicylate ligands of the small molecules with the particle surfaces.

The goal of this investigation was the fabrication of PAZO films by EPD, application of PAZO as a charging, dispersing and film forming agent for the EPD of various materials and investigation of properties of the films. The unique feature of the PAZO polymer is that salicylate ligands and aromatic groups of the individual monomers provide numerous adsorption sites for PAZO adsorption on the surfaces of inorganic particles (Fig. 1A(b)), carbon nanotubes and other materials and impart negative charges to the materials. We demonstrate superior performance of PAZO, compared to small organic molecules from the salicylic acid acid family. The enhanced adsorption and charging, coupled with improved steric stabilization resulted in good dispersion of different materials.

In this investigation we showed the possibility of EPD of PAZO films from different solvents, and proposed a deposition mechanism. The PAZO films, prepared by EPD, exhibited a photoinduced birefringence. The results presented below indicate that compared to other PAZO deposition techniques, EPD offers the advantages of simplicity, high deposition rate as well as excellent deposition rate control. The unique adsorption and film forming properties of PAZO allowed for the dispersion and EPD of various materials. As a proof of concept, we demonstrate that various functional materials of different types, such as TiO₂ (anatase and rutile), Al₂O₃ submicrometre particles and micron size platelets, zirconia, MnO₂, MgAl₂O₄, aluminium hydroxide, SiC, Pd metal, electrically neutral polypyrrole nanofibers and other polymers, carbon nanotubes, dyes and composites can be deposited using PAZO as a dispersing, charging and film forming agent. This versatility is a unique feature of PAZO and addresses the limitation of other common dispersing agents, that they are only capable of dispersing a few specific materials for EPD. The results of this investigation pave the way for the deposition of composite films utilizing the functional properties of different materials and PAZO. To this end, we investigated optical, charge storage, electrochemical and other properties of the composite films.

2. Experimental procedures

2.1. Materials

Poly[1-[4-(3-carboxy-4-hydroxyphenylazo)benzenesulfonamido]-1,2-ethanediyl, sodium salt] (PAZO) (Mw = 65,000–100,000, Aldrich, USA), poly(1-vinylpyrrolidone-co-2-dimethylaminoethyl methacrylate) (PVDM) (Mw = 1,000,000, Aldrich, USA), TiO₂ (anatase, <50 nm, Aldrich USA), TiO₂ (nanorods, rutile, <100 nm, Aldrich USA), submicrometre alumina (0.13 μ m, Baikowski, USA), alumina platelets (3–8 μ m, Elf Atochem, Canada), aluminium hydroxide (<1 μ m, Aldrich, USA), MgAl₂O₄ (-325 Mesh, Cerac, USA), zirconia (40 nm, Tosoh TZ-12CE, Japan), SiC (270 nm, Ibiden Co., Japan), multiwalled carbon nanotubes (MWCNT, ID 4 nm,

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