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Ethylene oxide-*block*-butylene oxide copolymer uptake by silicone hydrogel contact lens materials

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ABSTRACT

Four major types of silicone hydrogel contact lens material have been investigated following treatments in aqueous solutions containing poly(ethylene oxide) and poly(butylenes oxide) block copolymer (EO–BO). The extent of lens surface modification by EO–BO and the degree of bulk uptake were studied using X-ray photoelectron spectroscopy (XPS) and ultra-performance liquid chromatography (UPLC), respectively. The experimental results suggest that different interaction models exist for the lenses, highlighting the influence of both surface and bulk composition, which greatly differs between the lenses examined. Specifically, lenses with hydrophilic surface treatments, i.e., PureVision® (balafilcon A) and O_2OPTIX (lotrafilcon B), demonstrated strong evidence of preferential surface adsorption within the near-surface region. In comparison, surface adsorption on ACUVUE® Oasys® (senofilcon A) and Biofinity® (comfilcon A) was limited. As for bulk absorption, the amount of EO–BO uptake was the greatest for balafilcon A and comfilcon A, and least for lotrafilcon B. These findings confirm the presence of molecular concentration gradients within the silicone hydrogel lenses following exposure to EO–BO solutions, with the nature of such concentration gradients found to be lens-specific. Together, the results suggest opportunities for compositional modifications of lenses for improved performance via solution treatments containing surface-active agents.

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1. Introduction

Since their introduction, silicone hydrogel (SH) contact lenses have shown marked improvement over conventional hydrogel lenses by allowing extended periods of wear time without inducing corneal hypoxia. To promote wearability, SH lenses rely on siloxane-based polymers, rather than water, for oxygen transport through the bulk of the lens. From a materials science perspective, surface properties are also recognized to influence lens performance *in vivo*. This is a result of interactions involving the interfacial phenomena of wetting and friction between cornea, lens, and tear film. For SH lenses, the approach to enhanced oxygen transport can compromise the hydrophilic nature of the lens surface required for wetting by the tear film. This effect originates from the diffusion of hydrophobic siloxane moieties toward the lens surface (i.e., the air-lens interface), driven by the net thermodynamic stability of the system. Consequently, much development in SH lens materials design has focused on improving the *surface* hydrophilicity of the lenses via chemical (e.g., by manufacturer) and solution (e.g., by daily lens-care practices) treatments.

SH lenses are typically distinguished by their respective hydrophilic treatment. PureVision® (balafilcon A), Focus® NIGHT & DAY® (lotrafilcon A), and O_2OPTIX® (lotrafilcon B) undergo plasma treatments for enhanced surface hydrophilicity. Specifically, the surface of balafilcon A is treated with an oxygen plasma, resulting in changes of the oxidative state of the inherent silicon species. The lotrafilcon materials, on the other hand, receive a permanent, uniform surface treatment via plasma-induced polymerization. The incorporation of hydrophilic poly(vinylpyrrolidone) (PVP) into the bulk hydrogel enables lenses such as ACUVUE® Oasys® (senofilcon A) and ACUVUE® ADVANCE® (galyfilcon A) to obtain improved wetting behavior. The third strategy of improving lens surface hydrophilicity, represented by Biofinity® (comfilcon A), does not employ any surface treatment or wetting agent, but relies on specific silicone macromers constituting the hydrogel backbone to achieve wettability.

As in the case for most polymeric materials, surface properties of hydrogels are often dynamic in nature, heavily influenced by their environment and the measurement methodology. Examples of such behavior have been well documented in studies measuring the contact angles and friction response of hydrogel lens surfaces [1–3]. These results suggest that, in achieving thermodynamic equilibrium, changes in surface chemistry leading to different wetting and friction behaviors are inevitable. This is especially important for

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the performance of lenses in aqueous environment such as in the eye. Therefore, in addition to the hydrophilic treatments applied during manufacturing, treatments by solutions containing surface-active wetting agents (e.g., surfactants) are necessary to ensure proper wetting of the lens surface *in vivo*, providing comfort to patients who wear SH lenses for extended periods of time.

Past studies have analyzed changes in surface properties of hydrogel lenses following the adsorption of a variety of surfactant molecules including hydroxypropyl methylcellulose (HPMC) and poloxamine 1107 (Tetronic®, BASF) [1,4,5]. These studies concluded that the surface adsorption of these molecules resulted in improved wetting behavior of poly(2-hydroxyethyl methacrylate) (pHEMA) based lenses. Furthermore, subjects wearing the surfactant-treated lenses also reported enhanced comfort, underscoring the potential of this treatment to be used in clinical applications of conventional hydrogel lenses.

In addition to HPMC and poloxamine, small amphiphilic block copolymers have long been regarded as effective surface-active agents in a host of industrial and medical applications. Categorically, this nonionic and water-soluble class of molecules is especially suitable for modifying surfaces under aqueous conditions. As the reduction of friction and enhancement of comfort at the lens-tear film interface is a rising concern in the contact lens materials research field, amphiphilic block copolymers containing poly(ethylene oxide) (EO) as the hydrophilic component balanced by a relatively more hydrophobic component have received much attention as functional surface-active additives to multi-purpose disinfecting solutions (MPDS).

Much of the literature on EO-based amphiphilic block copolymers is devoted to the study of adsorption and association behaviors of poly(ethylene oxide)-poly(propylene oxide) (EO–PO) based triblock copolymers having various molecular architectures. The Pluronic® (BASF) series of block copolymers include various EO–PO-based copolymers, primarily used as anti-foaming agents in some lens-care solutions. It has been suggested, however, that the larger difference in polarity between the EO and the butylene oxide (BO) blocks in EO–BO copolymers can lead to greater surface activity, i.e., more effective surface tension reduction as compared to that observed with the structurally equivalent EO–PO copolymers [6,7].

Prior work within our group has characterized the interfacial friction of hydrated SH lenses in the presence of a poly(ethylene oxide)-*block*-poly(butylene oxide) (EO–BO) surfactant copolymer solution using atomic force microscopy (AFM) [8]. It was shown that in comparison to the neat lens samples, reduction in friction was apparent for a number of lenses exposed to this copolymer. Based on chemical modifications seen with X-ray photoelectron spectroscopy (XPS), surface adsorption of EO–BO was concluded. The extent of such modifications, however, was lens-dependent. Specifically, lenses having undergone surface plasma treatments, e.g., balafilcon A and lotrafilcon B, demonstrated much greater EO–BO adsorption, and consequently, friction reduction, in comparison to senofilcon A, which is not surface-treated during manufacturing. These preliminary results led to the speculation that the mechanism by which the surfactant molecules interact with different SH lenses is a function of each lens' surface chemistry, and such dependency can be probed by XPS, allowing semi-quantitative analysis of the adsorbed species [8]. In the former studies, the possibility of the absorption of the EO–BO copolymer into the bulk of SH lens was not precluded, thus prompting the quantitative investigation of copolymer net uptake. In the present study, the interaction mechanisms between EO–BO copolymers and surfaces of four types of SH lenses have been concurrently assessed by XPS and ultra-performance liquid chromatography (UPLC), respectively. The resulting correlation between the degree of surface *adsorption* and a quantitative measure of the amount of EO–BO copolymer uptake, reflective of bulk *absorption*, further reveals a rich disparity in the nature and degree of interaction between this surfactant and lenses of distinct compositions, as well as pathways to clinically relevant solution treatments.

2. Experimental

2.1. Materials

Four types of SH lenses were investigated: balafilcon A (PureVision®) by Bausch & Lomb, senofilcon A (ACUVUE® Oasys®) by VISTAKON of Johnson & Johnson, lotrafilcon B (O_2OPTIX®) by CIBA VISION®, and comfilcon A (Biofinity®) by CooperVision. The published composition and hydrophilic surface treatment of each lens are detailed in Table 1 [9–11]. Prior to analysis or solution treatment, all lenses were soaked in a standard buffered saline solution (Unisol®4, Alcon Inc., Fort Worth, TX) for at least 24 h. This procedure was necessary to remove residual components of packing solutions from the hydrogel surface [1,8]. Subsequent solution treatments involved soaking the lenses in solutions containing various EO–BO concentrations at room temperature for at least 24 h. All EO–BO treated lenses were analyzed without further solution treatment (e.g., rinsing) but were gently dried with tissue to remove excess solution. This procedure was employed to avoid the inadvertent condensation of EO–BO polymer from an evaporating final layer of solution. The diblock copolymer consisted of, on average, 45 units of ethylene oxide and 11 units of butylene oxide per chain.

2.2. Bulk uptake of EO–BO analyzed by UPLC

Bulk uptake of EO–BO was assessed using the extraction from each lens and analyzed by an UPLC system (Waters Corporation, Milford, MA, USA), which consisted of a binary solvent separation module and a charged aerosol universal detector (CAD). The separation was performed on an ACQUITY UPLC™ column (2.1 mm × 150 mm, 1.7 μm particle size) with a mobile phase consisting of methanol (solvent A) and 0.1% trifluoroacetic acid/diamond water (solvent B). A semi-gradient chromatographic condition was applied to the mobile phase in order to establish an acceptable baseline for the elution study. Subsequently, standards of EO–BO and EO–BO extract dissolved in methanol from each treated lens were acquired and analyzed using Empower software (Waters Corporation, Milford, MA, USA), resulting in the amount of EO–BO uptake measured as a function of retention time.

2.3. Surface chemical composition analyzed by XPS

A commercial XPS system (Omicron NanoTechnology, Taunusstein, Germany) with a monochromatic Al source (1486.7 eV) and a hemispherical analyzer EIS-Sphera (Omicron NanoTechnology, Taunusstein, Germany) was used to measure surface composition. The lenses in this study were mounted and dried in 10^{-7} Torr vacuum for about 12 h before conducting each XPS measurement, which took place in a 2×10^{-10} Torr vacuum environment. Core-level XPS scans of the primary elements were acquired at a pass energy of 20 eV and a step size of 0.05 eV. Charge neutralization was employed by compensating the sample surface with both low energy electrons and low energy ions during data collection. This procedure reduced the charging effects generally observed in insulating samples and improved energy resolution of the respective peaks [12]. The surface sensitivity of the measurements was determined by the take-off angle (TOA) of the instrument, which is defined as the angle between the surface normal and the analyzer.

The spectroscopic data were processed using CasaXPS software (Casa Software Ltd.). Peaks were assigned with the aid of the

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