# Imparting Antifouling Properties of Silicone Hydrogels by Grafting Poly(ethylene glycol) Methyl Ether Acrylate Initiated by UV Light

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Received 12 March 2011; accepted 20 September 2011
DOI 10.1002/app.35687
Published online 22 December 2011 in Wiley Online Library (wileyonlinelibrary.com).

**ABSTRACT:** Both hydrophilic and antifouling surfaces were prepared on silicone hydrogels with poly(ethylene glycol) methyl ether acrylate (PEGMA) grafted by UV-induced radical polymerization. The PEGMA-grafted silicone hydrogels were characterized by graft yield and static water contact angle measurements. According to the results, the graft yield reached a maximum at 8 min of UV exposure time and 20 wt% PEGMA concentration. The modified silicone hydrogels possessed hydrophilic surfaces with the lowest water contact angle of 36°. The oxygen permeability and transparency of the PEGMA-grafted silicone hydrogels were as high as the unmodified silicone hydrogel. The mechanical property of silicone hydrogels was maintained at about 95% of the tensile strength and elastic modulus after the PEGMA grafting. The *in vitro* single protein adsorption on the PEGMA-grafted silicone hydrogels decreased by 70–80% compared to the unmodified silicone hydrogel. The PEGMA-grafted silicone hydrogel is expected to be a novel biomaterial, which possesses excellent surface hydrophilicity, antifouling property, oxygen permeability, and mechanical property. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 125: 548–554, 2012

**Key words:** silicone hydrogel; poly(ethylene glycol) methyl ether acrylate (PEGMA); grafting; antifouling

## INTRODUCTION

Silicone hydrogels, which have attracted increasing attention as a new generation of soft contact lens, lead to great advance in the field of vision correction.[1] Contact lenses made from these materials satisfy the metabolic needs of the cornea and can be worn continuously for up to 30 days.

However, the native hydrophobicity and biofouling tendency of silicone hydrogels have been one of the biggest limitations for biomaterial applications. To modify the disadvantages of silicone hydrogel-based materials, surface coating or grafting with hydrophilic polymers on silicone hydrogels is the most widespread technique to improve the surface hydrophilicity and antibiofouling property. Surface graft polymerization is better than coating because of the chemical stability of its covalent bonding with a substrate and lower risk for deposition.[2] It may be anticipated that the hydrophilicity is responsible for less protein adsorption. The protein repellent properties would prevent an extracellular matrix from being formed, resulting in improved biocompatibility.

The conventional grafting polymerization technique requires chemically reactive groups on the surface. For this reason, a series of functionalization techniques, such as ultraviolet (UV) irradiation,[3–6] plasma,[7,8] ion beams,[9] and chemical initiators,[10,11] are necessary for covalent grafting. Among these techniques, the radiation grafting method is one of the most preferable methods because of its uniform and rapid generation of active radical sites without catalytic contamination on the surface of grafting materials.

UV radiation grafting methods, including preirradiation and simultaneous irradiation grafting, can introduce specific functional moieties to a polymeric substrate. For the former method, the polymeric substrate is irradiated first in vacuum, nitrogen or air, and then the subsequent monomer is grafted by peroxide radicals; for the latter method, the polymer substrate is simultaneously irradiated in the presence of monomers. Consequently, when vinyl monomers are present, free-radical graft polymerization occurs at these reactive sites, resulting in the formation of

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Contract grant sponsor: Natural Science Fund for Colleges and Universities; contract grant number: 11KJB430013.
Contract grant sponsor: Natural Research Fund; contract grant number: XKR2011010.
Contract grant sponsor: Research Fund of Key Laboratory for Advanced Technology in Environmental Protection of Jiangsu Province; contract grant number: AE201069.

polymer chains that are covalently bonded to the surface of the substrate.[4]

The nonfouling surfaces can be prepared by the grafting of neutral and water-soluble polymers, such as poly(acrylamide), poly(2-hydroxyethyl methacrylate), poly(*N*, *N*-dimethylacrylamide), poly(2-methacryloxloxyethyl phosphorylcholine), and poly(ethylene glycol) (PEG).[12] In the previous report, 2-methacryloyloxyethyl phosphorylcholine (MPC), which can be regarded as a biomimetic component of cell membrane, was tethered onto the silicone hydrogels through air plasma pretreatment and then heat induced graft polymerization to improve the surface biocompatibility. However, the complicated procedures can increase overall manufacturing costs.[13–15] The most effective polymer for preparing protein-repellent surfaces appears to be PEG, which is a nontoxic, nonimmunogenic, and nonantigenic polymer. PEG is known to decrease the attractive forces between surfaces and proteins as a result of high mobility in the hydrated state and related steric repulsion.[16–18] The covalent grafting of PEG by plasma and chemical initiators onto a variety of substrates, including silicon,[3,6,19] polyurethane,[20] fluorinated ethylene-propylene copolymers,[21] polysulfone membranes,[22] and hydrogels,[1,10,11] has been reported along with a quite satisfactory protein-repellent effect. In addition to these antifouling properties, PEG is optically transparent when hydrated, which is of the utmost importance for materials to be used as optical devices.[11] To our knowledge, there are until now no reports of UV-induced graft of PEGMA onto silicone hydrogels for the development of biomaterials with enhanced hydrophilicity and protein resistance.

In this report, silicone hydrogels were prepared by the copolymerization of methacrylated polydimethylsiloxane macromonomer, hydrophobic comonomer 3-methacryloxypropyl tris(trimethylsoloxy silane) (TRIS), and hydrophilic comonomer *N*,*N*-dimethyl acrylamide (DMA). Then the obtained silicone hydrogels were modified by simultaneous irradiation grafting poly(ethylene glycol) methyl ether acrylate (PEGMA) initiated by UV light, which was regarded as the most convenient and efficient method. The PEGMA-grafted silicone hydrogels were characterized by graft yield and static water contact angle (SCA) measurements. Eventually, the oxygen permeability, mechanical properties, and protein adsorption of the PEGMA-grafted silicone hydrogels were investigated.

## EXPERIMENTAL

### Materials and methods

Methacryloxypropyl tris (trimethylsiloxy) silane (TRIS) and *N*,*N*-dimethylacrylamide (DMA) were purchased from Aldrich and purified by distillation under reduced pressure before use. Free radical photoinitiator Darocur 1173 was obtained from Ciba Co. Poly(ethylene glycol) methyl ether acrylate (PEGMA, number average molecular weight 320) from Aldrich was the analytical-grade product and used as received. 1,1-Diphenyl-2-picrylhydrazyl (DPPH) obtained from Aldrich was used for the quantification of peroxide concentration produced from UV irradiation. Albumin, fibrinogen, and lysozyme were purchased from Biozyme Company. The bifunctional methacrylated polydimethylsiloxanes macromer (MS) was prepared by the method as described in the literature.[23]

### Preparation of silicone hydrogels

Bifunctional methacrylated polydimethylsiloxanes macromer (MS, 35 wt%), TRIS (35 wt%), and DMA (30 wt%), in a weight ratio chosen to total 100 parts, were mixed with 20 parts of *n*-hexanol and 0.5 part of photoinitiator Darocur 1173.[2] The mixture was introduced between two glass plates (7.5 × 2.5 $cm^2$) and cured for 1 h under a high-pressure mercury lamp emitting UV light with a mixed wavelength, where the main wavelengths were 305 and 365 nm. Film thickness was controlled by a Teflon gasket, which gave a fairly consistent thickness of 0.25 mm. The films were, respectively, extracted with ethanol and water for 24 h. After immersing in fresh distilled water, transparent silicone hydrogel membrane was obtained with water content of 31 wt% measured by gravimetric assay.

### Surface modification of silicone hydrogels by UV irradiation

The grafting experiment was carried out by a simultaneous irradiation grafting method. PEGMA/ethanol solution was purged with nitrogen for 2 h to minimize the presence of oxygen. Subsequently, the PEGMA/ethanol solution (5, 10, 15, 20, and 25 wt%) was introduced into a slit constructed between a silicone hydrogel membrane and a photomask by utilizing cover glasses (150-μm thickness, Matsunami Glass) as spacers and was then exposed through the photomask to UV light with a mixed wavelength, where the main wavelengths were 305 and 365 nm. The cover glasses were used as transparent photomasks, and the UV light was irradiated through the cover glasses to obtain completely grafted samples. After UV irradiation for a prescribed time, the silicone hydrogel membrane was rinsed with deionized water for 1 day to remove unreacted PEGMA and homopolymer of PEGMA. Finally, PEGMA-grafted silicone hydrogels were obtained and stored in water.[4,6,24]

### Determination of UV irradiation time

To obtain the optimal UV irradiation time, the amount of peroxide formed around the membrane after UV irradiation was quantified by the DPPH titration method.[25] The DPPH/toluene solution was degassed by a nitrogen gas purge for 30 min. The irradiated silicone hydrogel membrane was dipped into the DPPH/toluene solution at 65°C for 2 h in a shaking water bath to decompose the peroxides formed around the membrane. The amount of DPPH reacted with peroxides was measured with a JASCO V-560 UV/vis spectrophotometer at 520 nm. A calibration curve was obtained with five DPPH/toluene solutions of known concentration (0.0625, 0.125, 0.25, 0.5, and $1 \times 10^{-4}$ mol/L). To obtain the optimal UV irradiation time, the relationship between irradiation time and peroxide concentration was investigated.

### Characterization of PEGMA-grafted silicone hydrogels

The graft yield of samples was obtained gravimetrically by the following equation:

$$\text{Graft yield } (\text{mg/cm}^2) = (W_f - W_i) \div A$$

where $A$ was the surface area of silicone hydrogel membrane, $W_f$ and $W_i$ were the final and initial weights of silicone hydrogel membrane before and after UV-induced grafting modification, respectively.

The SCA of samples was measured at ambient humidity and temperature by the sessile drop method, using JC2000C1 goniometer of Zhongchen Digital Technical Co., China. The contact angle reported here was an averaged value of at least three measurements.

The oxygen permeability of samples was measured by the two-chamber method[26] on a Mocon OX-TRAN® model 2/21 oxygen transmission rate tester and was expressed as Dk in unit of barrer (1 barrer = $10^{-11}$ $\text{cm}^2$ mL $O_2$/s mL mmHg).

Thickness of samples was measured by ellipsometry. Measurements were taken on a Gaertner LSE single wavelength ellipsometer. The light source had an angle of incidence of 70° and was generated by a helium-neon laser. Nine measurements were taken per substrate and averaged to give the thickness grafted to the silicone hydrogel substrate. Averaging thickness of three substrates gave the average thickness reported for each sample.

Mechanical properties of samples were carried out using an Instron series IX materials testing system at room temperature. Dog-bone shaped samples were cut from the hydrogels (5-mm wide at the narrowest point with a gage length of 15 mm). Thickness of the samples was measured with a digital micrometer having a precision of 1 μm. A crosshead speed of 10 mm/min was used and at least triplicate was tested for each sample.

The transparency of samples was examined by using JASCO V-560 UV/vis spectrophotometer. The measurements were performed from 400 to 800 nm wavelength at room temperature.

The surface elemental composition of samples at dry state was analyzed by X-ray photoelectron spectroscopy using a Shimadzu ESCA 750 spectrometer using MgKa radiation. The take-off angle of photoelectron was 45°.

*In vitro* single protein adsorption experiments were performed in phosphate-buffered saline (PBS, pH 7.4). Samples were immersed in 4.5 mg/mL of bovine serum albumin, 0.3 mg/mL of fibrinogen from bovine serum, and 2.0 mg/mL of lysozyme from chicken egg white solutions, respectively. Modified and unmodified silicone hydrogel membranes were first immersed in PBS-filled 24-well plate for 24 h to be full hydrated. The samples were moved into wells containing single protein solutions, and adsorptions were allowed to proceed at 37°C for 12 h under gentle shaking. Each sample was then rinsed in the fresh PBS by 50 dippings. The samples were subsequently transferred into a well-plate filled in 1 mL of PBS solution containing 1 wt% of sodium dodecyl sulfate (SDS), and the adsorbed protein was completely desorbed by sonication for 5 h. The concentration of protein in the SDS solution was determined by the bicinchoninic acid assay method.[5] From the concentration of protein, the amount of protein adsorbed on the surface was calculated.

## RESULTS AND DISCUSSION

### Determination of UV irradiation time

The peroxide species can be used to initiate the surface free-radical polymerization in a mechanism generally proposed for the UV-induced surface graft polymerization.[12] Free radicals generated on the original silicone hydrogel membrane without dipping into the PEGMA monomer solution after UV irradiation could be used to initiate the grafting reaction. The amount of peroxide was determined by measurement of the concentration of consumed DPPH molecules that reacted with free radicals generated after UV irradiation. Therefore, the DPPH titration method was utilized to determine the optimal UV irradiation time.[4] Figure 1 shows the effect of UV irradiation time on the radical concentration generated on the silicone hydrogel surface after UV irradiation. Seen from Figure 1, the concentration of peroxides increased with the UV irradiation time and reached almost constant at 8 min. According to the results, an exposure

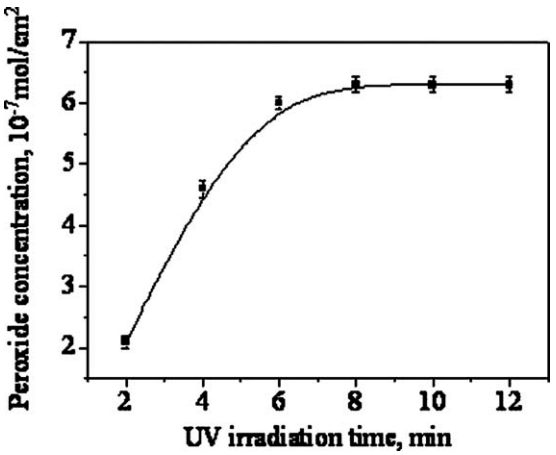


**Figure 1** Effect of UV irradiation time on the peroxide concentration generated on the silicone hydrogel surface after UV irradiation.

time of 8 min was regarded as the optimal UV irradiation time for this study.

### PEGMA graft polymerization on silicone hydrogels

The grafting experiment was carried out by a simultaneous irradiation grafting method. And the process is shown schematically in Figure 2. The effect of PEGMA concentration on the graft yield and wettability was investigated with irradiation time of 8 min. As shown in Figure 3, in the case of PEGMA-grafted silicone hydrogels, as the graft yield increased with the PEGMA concentration, the SCA of membranes decreased because of the hydrophilicity of the grafted PEGMA. However, above the concentration of 20 wt%, the SCA gradually increased. It was deduced that excessive use of the PEGMA monomer accelerated the formation of homopolymer of PEGMA, without reaction with the substrate of the silicone hydrogel.[4] The PEGMA-grafted silicone hydrogels abbreviated as Si-*g*-PEGMA 1 to 4 were obtained, respectively, by graft polymerization of PEGMA with concentrations of 5, 10, 15, and 20 wt%, fixing the irradiation time of 8 min. For comparison, Si-*g*-0 referred to the pristine unmodified silicone hydrogel.

### Oxygen permeability

Oxygen permeability is an important factor for biomaterial applications, such as ophthalmologic biomaterials, and artificial lungs. Therefore, the effect of the PEGMA grafting on the oxygen permeability was checked by directly measuring the Dk values of PEGMA-grafted silicone hydrogels. The expression Dk, which is the product of the oxygen diffusion coefficient (D) and the oxygen solubility coefficient in the material (k), has become universally accepted, as the term referring to the intrinsic property of a material to transport oxygen through its bulk.

Table I lists the graft layer thickness and Dk values of the PEGMA-grafted silicone hydrogels. The DK of the unmodified silicone hydrogel was 143.5 barrer and that of the PEGMA-grafted silicone hydrogels ranged from 142.0 to 143.2 barrer. The PEGMA-grafted silicone hydrogels maintained about 99% of the oxygen permeability due to its considerably thinner graft layer thickness (less than 100 nm) compared to that of the silicone hydrogel membrane (0.25 mm).

The oxygen permeability was extensively discussed in the field of soft contact lens biomaterials. It was suggested that the oxygen transmittance, Dk/L (thickness of contact lens), for continuous wear soft contact lens should be 87–125 barrer/mm for a human cornea. Considering the commercial soft contact lens thickness of less than 100 μm, this oxygen transmission requirement was easily obtained for the PEGMA-grafted silicone hydrogels in this research.[5]

### Mechanical property

The UV-induced free-radical graft polymerization methods may pose a problem concerning the bulk mechanical property for the silicone hydrogels. Tensile tests were performed on the PEGMA-grafted

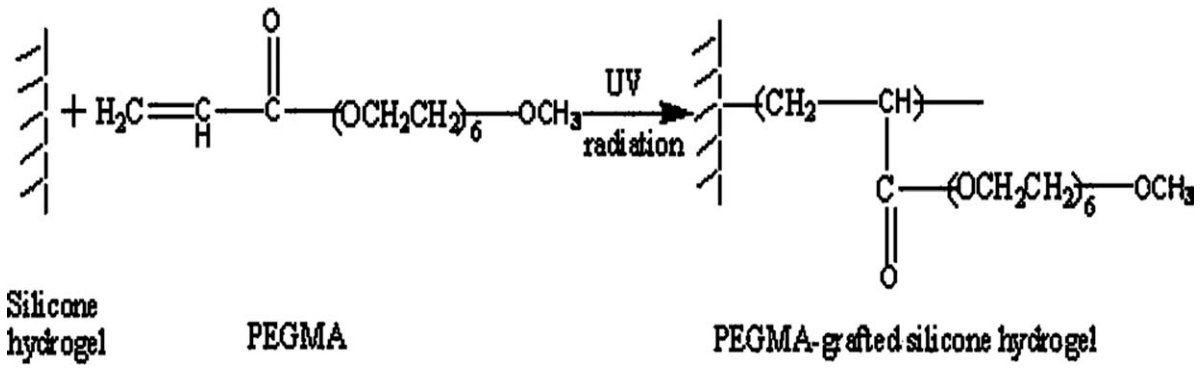


**Figure 2** Schematic diagram illustrating the modification processes of the silicone hydrogel by UV-induced graft polymerization.

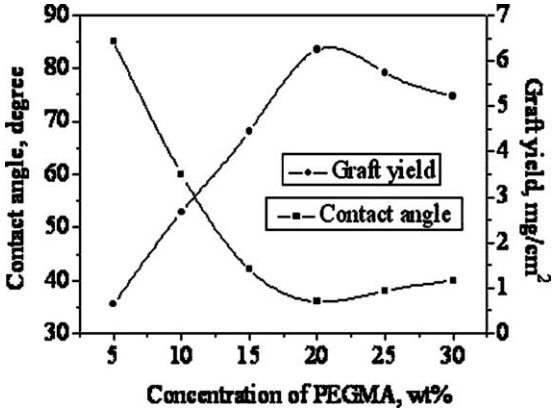


**Figure 3** Effect of the PEGMA concentration on the graft yield and wettability.

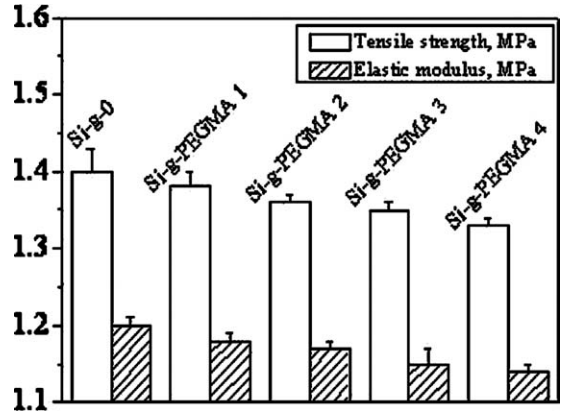


**Figure 4** Mechanical properties of the unmodified and PEGMA-grafted silicone hydrogels.

silicone hydrogels to determine the effect for the bulk phase of silicone hydrogels.

Figure 4 shows the results of the tensile strength and elastic modulus for the unmodified and PEGMA-grafted silicone hydrogels. Both the tensile strength and elastic modulus of the PEGMA-grafted silicone hydrogels were lower than those of the unmodified silicone hydrogels by less than 5%. Seen from the results, although the deterioration of the silicone hydrogel bulk properties occurred, it seemed to be too limited to affect the practical uses of silicone hydrogels. These results also agreed with a previous report on the grafting of MPC on polydimethylsiloxane (PDMS) surfaces by photo-induced radical polymerization.[5]

## Optical transparency

It is of critical importance that the transparency of the silicone hydrogels is maintained after the PEGMA grafting, if the silicone hydrogels are used as contact lens. Figure 5 shows the transparency of the unmodified and PEGMA-grafted silicone hydrogels measured by UV spectrophotometry in a wavelength range between 400 and 800 nm. Clearly, grafted or not by PEGMA, the visible light transmission by the silicone hydrogels was all higher than 92%. The transparency of the silicone hydrogels was thus unaffected by the PEGMA grafting. The PEGMA-grafted silicone hydrogels were visually transparent all the same, which was confirmed by transparency measurements, as shown in Figure 5. The results were in accordance with a previous study in which the visible light transmission by the hydrogel intraocular lens was higher than 90%.

**TABLE I**
**DK Values and Graft Layer Thickness of the PEGMA-Grafted Silicone Hydrogels**

| Sample | DK (barrer) | Graft layer thickness (nm) |
|---|---|---|
| Si-*g*-0 | 143.5 ± 0.1 | 0 |
| Si-*g*-PEGMA 1 | 143.2 ± 0.1 | 32 ± 1.9 |
| Si-*g*-PEGMA 2 | 142.8 ± 0.2 | 55 ± 1.2 |
| Si-*g*-PEGMA 3 | 142.3 ± 0.1 | 75 ± 1.5 |
| Si-*g*-PEGMA 4 | 142.0 ± 0.2 | 82 ± 2.1 |

## XPS analysis

X-ray photoelectron spectroscopy (XPS) was used to characterize the surface of PEGMA-modified and unmodified silicone hydrogels. The main reason for the use of XPS was the information depth of this analytical method (~ 10 nm for polymeric materials), and the ability of this method to obtain comprehensive information about the elemental and chemical composition of samples in a single experiment.[1,2]

Table II shows the elemental compositions of PEGMA-grafted silicone hydrogel surfaces determined by XPS at the take off angle of 45°. With the increase of the graft yield, the compositions of C and O increased, whereas the composition of Si decreased.

The C/O ratios of the PEGMA-grafted silicone hydrogel surfaces were close to the theoretical 2/1

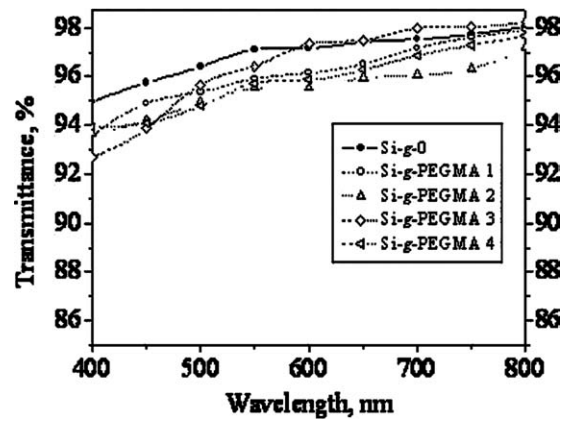


**Figure 5** Transmittance curves of unmodified and PEGMA-grafted silicone hydrogels.

**TABLE II**
**Surface Elemental Compositions of the PEGMA-Grafted Silicone Hydrogels Determined by XPS at a 45° Take Off Angle**

| Sample | C | O | N | Si | C/O |
|---|---|---|---|---|---|
| Si-*g*-0 | 58.3 | 26.5 | 2.1 | 13.1 | 2.20 |
| Si-*g*-PEGMA 1 | 60.9 | 30.3 | 1.4 | 7.4 | 2.01 |
| Si-*g*-PEGMA 2 | 62.9 | 31.2 | 1.0 | 4.9 | 2.02 |
| Si-*g*-PEGMA 3 | 63.9 | 31.8 | 0.8 | 3.5 | 2.01 |
| Si-*g*-PEGMA 4 | 65.1 | 32.4 | 0.5 | 2.0 | 2.01 |

ratio calculated from the repeat unit ($CH_2CH_2O$) of PEGMA. The composition of Si was lower in the PEGMA-grafted silicone hydrogel surfaces than in the unmodified silicone hydrogel, indicating that both of the repeat unit ($CH_2CH_2O$) and $(CH_3)_2SiO$ were present on the PEGMA-grafted silicone hydrogel surfaces, although the amount of the latter decreased as the graft yield increased.

### *In vitro* protein adsorption

Proteins contained in the eye liquids in contact with the implanted lenses are expected to adsorb very rapidly onto the surface of the lenses. This nonspecific adsorption of proteins is uncontrolled and is thought to trigger deleterious reactions of the body, such as foreign body response and fibrous encapsulation. Therefore, *in vitro* protein adsorptions were tested to estimate the protein repellency properties of the coated silicone hydrogels in relation with the graft yield.[2,5]

Figure 6 shows the amounts of albumin, fibrinogen, and lysozyme adsorption on the silicone hydrogel membranes. There were little differences in the amount of albumin adsorption among the PEGMA-grafted silicone hydrogel membranes. The average amount of albumin adsorption on the PEGMA-grafted silicone hydrogel surfaces was 0.40 μg/cm$^2$, which was about an 80% reduction compared to the unmodified silicone hydrogel. As for fibrinogen and lysozyme, the average adsorption amounts on the PEGMA-grafted silicone hydrogel membranes were 0.6 and 0.5 μg/cm$^2$, which were, respectively, 75% and 70% reductions compared with the unmodified silicone hydrogel. Similar results have also been reported by Sugiura and coworkers. They applied photo-induced graft polymerization to micropatterned surface modification of PDMS with PEGMA. The PEGDA-grafted PDMS exhibited useful characteristics (e.g., hydrophilicity, low protein adsorption, and low cell attachment) for various microfluidic devices.[6]

The reduction in protein adsorption to the silicone hydrogel surface modified by the surface graft polymerization must be due to the hydrated graft chains tethered onto the silicone hydrogel surface. The graft polymer chains at this diffuse interface may prevent protein molecules from their direct contact with the silicone hydrogel surface because of their steric hindrance effect. It should be pointed out that the grafted surface is different in nature from the conventional hydrophilic surfaces such as poly(2-hydroxylethyl methacrylate), which may adsorb proteins directly through ionic, hydrogen bonding, or van der Waals force. The diffuse layer formed by water-soluble, nonionic graft chains has a potential to suppress the direct contact of proteins with the substrate surface.

### Storage stability of the grafted layer

The PEGMA-grafted silicone hydrogels were kept in water, and changes in contact angle examined as a function of time after the UV-induced graft polymerization are plotted in Figure 7. It was found that the silicone hydrogels grafted with hydrophilic monomer PEGMA exhibited almost the same contact angle during storage. The result was the same as the

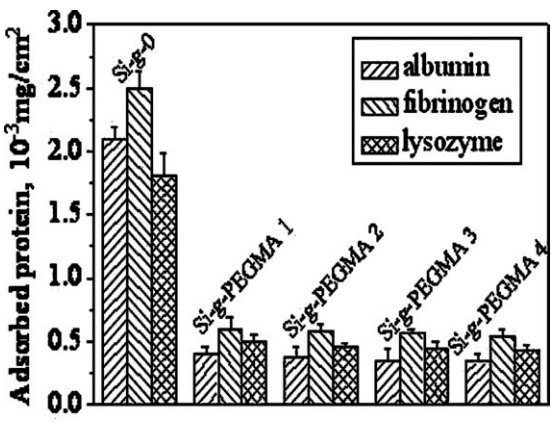


**Figure 6** Amount of single protein adsorption on the unmodified and PEGMA-grafted silicone hydrogels.

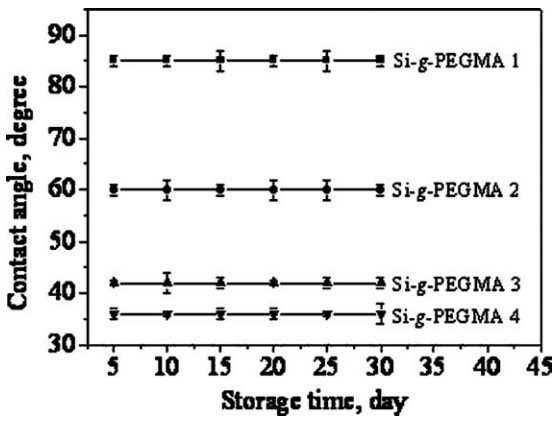


**Figure 7** Change of the water contact angles of PEGMA-grafted silicone hydrogels with time after being stored in water.

study launched by sun et al.[2] It was indicated that covalently introduced PEGMA caused the stable surface hydrophilicity.

## CONCLUSIONS

In this report, PEGMA was grafted onto silicone hydrogels by UV-induced radical polymerization, to enhance the surface hydrophilicity and antifouling property. The results indicated that the PEGMA-grafted silicone hydrogels possessed hydrophilic surfaces with the lowest water contact angle of 36°. The high oxygen permeability, transparency and mechanical property of the unmodified silicone hydrogel were maintained after the PEGMA grafting. Reductions of the adsorbed single proteins on the PEGMA-grafted silicone hydrogels were about 70–80%. The PEGMA-grafted silicone hydrogels is attractive for biomedical materials especially contact lens.

The authors express their gratitude to the education department of Jiangsu Province and the Yancheng Institute of Technology.

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