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ARTICLE

Smart tailoring of the surface chemistry in GPTMS hybrid organic-inorganic films

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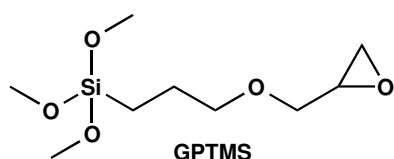
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Hybrid films prepared from 3-glycidoxypropyltrimethoxysilane have been widely used as organic-inorganic materials for several applications. Tailoring the coating surface should disclose new possibilities of applications in biomaterials as functional interfaces for cells and enzymes. In this work we have designed the synthesis of 3-glycidoxypropyltrimethoxysilane hybrid films to modify the surface properties without additional surface functionalization steps. The pH of the precursor sols has been changed from highly basic to acidic and neutral pH and then the sols have been used to deposit highly transparent films. The analysis by infrared spectroscopy has shown that the synthetic conditions allow tuning the degree of condensation of the silica network and the percentage of epoxide ring opening. A precise control of these two parameters enables the formation of a smart surface library where hydroxyl or epoxide groups or mixed presence of both change the hydrophobicity of the surface and thus its capability of binding molecules and nano-objects.

Introduction

3-Glycidoxypropyltrimethoxysilane (GPTMS) (**Scheme 1**) is one of the most used organically modified alkoxides for the synthesis of hybrid organic-inorganic materials;^{1,2} however the chemistry of GPTMS remains complex because organic and inorganic condensation reactions simultaneously take place during the sol to gel transition. In addition, several side reactions can also occur during the synthesis, making the complete control on the whole process an hard task to achieve.³ As a consequence, small changes in the chemistry of the precursor sol produce hybrid materials with different properties and structure.⁴



Scheme 1. Molecular structure of 3-Glycidoxypropyltrimethoxysilane (**GPTMS**).

One important feature of GPTMS is the presence of an epoxy-ring at the end of the propyl-chain. This functional group allows obtaining several chemical structures depending on the reactivity induced by different chemical surroundings. For instance, the reaction between GPTMS epoxy-ring and amines can be used to produce curing agents commonly used to

fabricate a wide class of thermosetting organics or as a coupling agent to covalently bind organic and inorganic networks.^{5,6}

In previous works we have extensively explored the sol-gel chemistry of GPTMS.⁷ In particular, when the reaction is carried out in conditions of extreme basic pH, we have found that self-assembled nano-crystals form both in bulk and films. The synthesis can be considered solventless since it is realized by adding less than a third of a sodium hydroxide aqueous solution to two thirds of neat GPTMS; optically transparent films of controlled thickness can be obtained even when hybrid nanocrystals form inside the material.

When the epoxide is opened by hydroxyl groups, it forms diols that further condense in ethylene oxide chains of different length or bridged silsesquioxane species. If GPTMS reactions take place in high pH conditions, both the epoxide ring and the silica condensation reactions are strongly slowed down. This favours the formation of bridged species *via* reaction of the epoxide that forms dioxane bridging species. In this case layered hybrid crystals are formed through molecule-by-molecule self-organization, and their alignment gives rise to optical anisotropy and birefringence. These are quite unique conditions if we consider that, in general, formation of crystals through self-organization is observed only when bridged silsesquioxanes are used.⁸

There exists a mutual dependence between the formation of the inorganic network, through silanol polycondensation, and the organic chain length, which forms upon opening of the epoxides. If the inorganic backbone forms too quickly, the

organic moieties do not have enough time to fully polymerise and tend to form only oligo-species. This means that the capability of controlling the organic and inorganic reactions is crucial to tune the property of the final material.

The chemistry of the epoxides in GPTMS affects not only the bulk, but also the surface properties; for instance a different hydrophilicity of the hybrid surface is expected depending on the number of closed epoxides. This property has been recently exploited to design smart surfaces for controlled drug release; GPTMS based hybrid films have been successfully used to bind ceria nanoparticles and release them in aqueous buffer solution within a time scale of hours.⁹

In the present work we have therefore investigated the GPTMS reactivity in a variety of pH conditions to tune the surface properties of the resulting matrix. This study is of peculiar importance for producing both effective hybrid interfaces with binding capabilities towards organic species, such as cells or biopolymers, and controlled grafting of nanosystems.

Experimental

Chemicals. 3-Glycidoxypropyltrimethoxysilane (GPTMS) (Sigma, >99%), ethanol (Sigma-Aldrich, > 99,8%), NaOH (Sigma-Aldrich, 2M) and HCl (Sigma-Aldrich, 2M), PBS 10x (Sigma-Aldrich, Phosphate Buffered Saline 10X Concentrate). Silicon wafers (Si-Mat) (100) cut, *p*-type boron doped, 350 μm thick, and silica slides (Haereus) were used as substrates for film deposition.

Synthesis of the precursor sols and film deposition. Two sols were prepared by mixing GPTMS with NaOH or PBS 1X respectively. The first sol was obtained by mixing 5 cm^3 of GPTMS (5.350 g, 22.64 mmol, 1 eq.) with 2 cm^3 of 2M aqueous NaOH (0.16 g, 4 mmol, 0.18 eq. of NaOH and 1.84g, 102.22 mmol and 4.52 eq. of H_2O) in a glass vial. The H_2O /GPTMS molar ratio into the fresh made solution is therefore equal to 4.52. The solution was left to react for 30 minutes after which the vial was closed with a screw cap and left under stirring for 12 h.

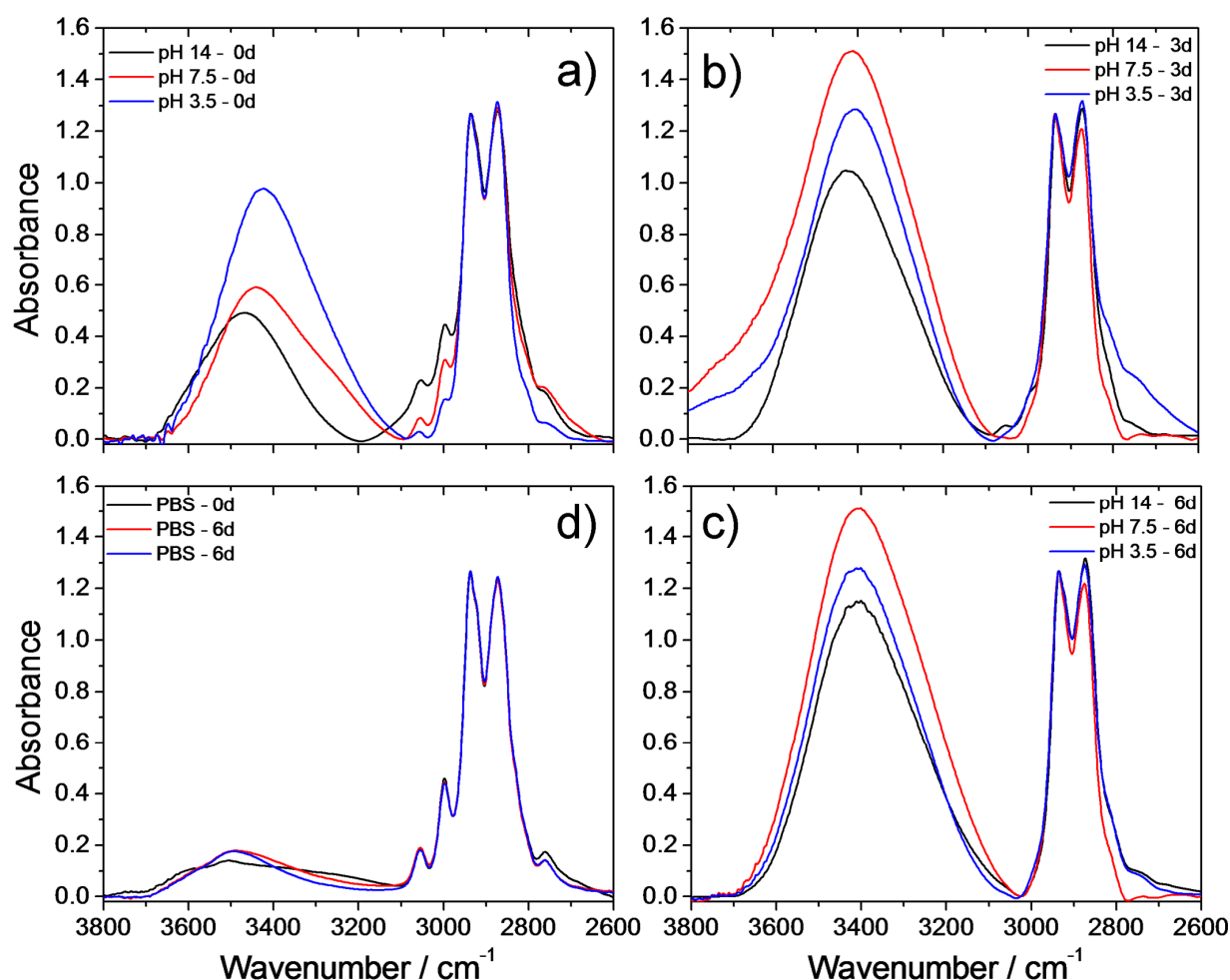


Figure 1. FTIR absorption spectra in the 3800 - 2600 cm^{-1} range of the films prepared at different pH conditions (black line for pH 14, red line for pH 7.5 and blue line for pH 3.5) of the precursor sol and aging time, 0 (a), 3 (b) and 6 days (c). The spectra of the films prepared by adding PBS buffer at different aging times are shown in (d), (black line for aging 0 days, red line for aging 3 days and blue line for aging 6 days). The spectra were normalised to the peak of CH_2 stretching at 2937 cm^{-1} to allow quantitative comparisons.

The resulting hybrids sol was then divided in 3 vials and the pH adjusted by adding dropwise either aqueous HCl or NaOH until the desired value was achieved. The second sol was prepared by adding 2 cm³ of PBS 1X to 5 cm³ of GPTMS (5.350 g, 22.64 mmol, 1 eq.). The resulting biphasic solution was left under stirring for 12 h until a complete homogenous solution was achieved. Afterwards, the sols were aged for up to 6 days in sealed vials and hybrid films were deposited from 0, 3 and 6 day aged sols. The films were spin-coated onto silicon wafers and silica slides at 5000 rpm for 20 seconds. The depositions were performed at room temperature and controlled relative humidity (< 50%). A particular care was taken to keep the relative humidity as constant as possible during spin-coating. Afterwards, the films were placed in an oven at 100°C for 1h in air at room pressure.

Materials characterization. Fourier transform infrared (FTIR) spectroscopy analysis was performed by a Bruker infrared Vertex 70V interferometer. The spectra were recorded on the films deposited on silicon in transmission mode, in the 400–4000 cm⁻¹ range by averaging 256 scans with 4 cm⁻¹ of resolution. The spectra of neat GPTMS were measured by casting a droplet of the liquid precursor on silicon wafer. The background was evaluated by measuring the signal of a clean silicon wafer substrate; the baseline was fitted by a concave rubber band correction with OPUS 7.0 software.

A Nicolet Evolution 300 UV-Vis spectrometer was used to acquire spectra in the 200-800 nm range using a variable step size resolution of the monochromator (intelliscan mode) with an integration time of 2 s per step. A silica slide was used to correct the background absorption.

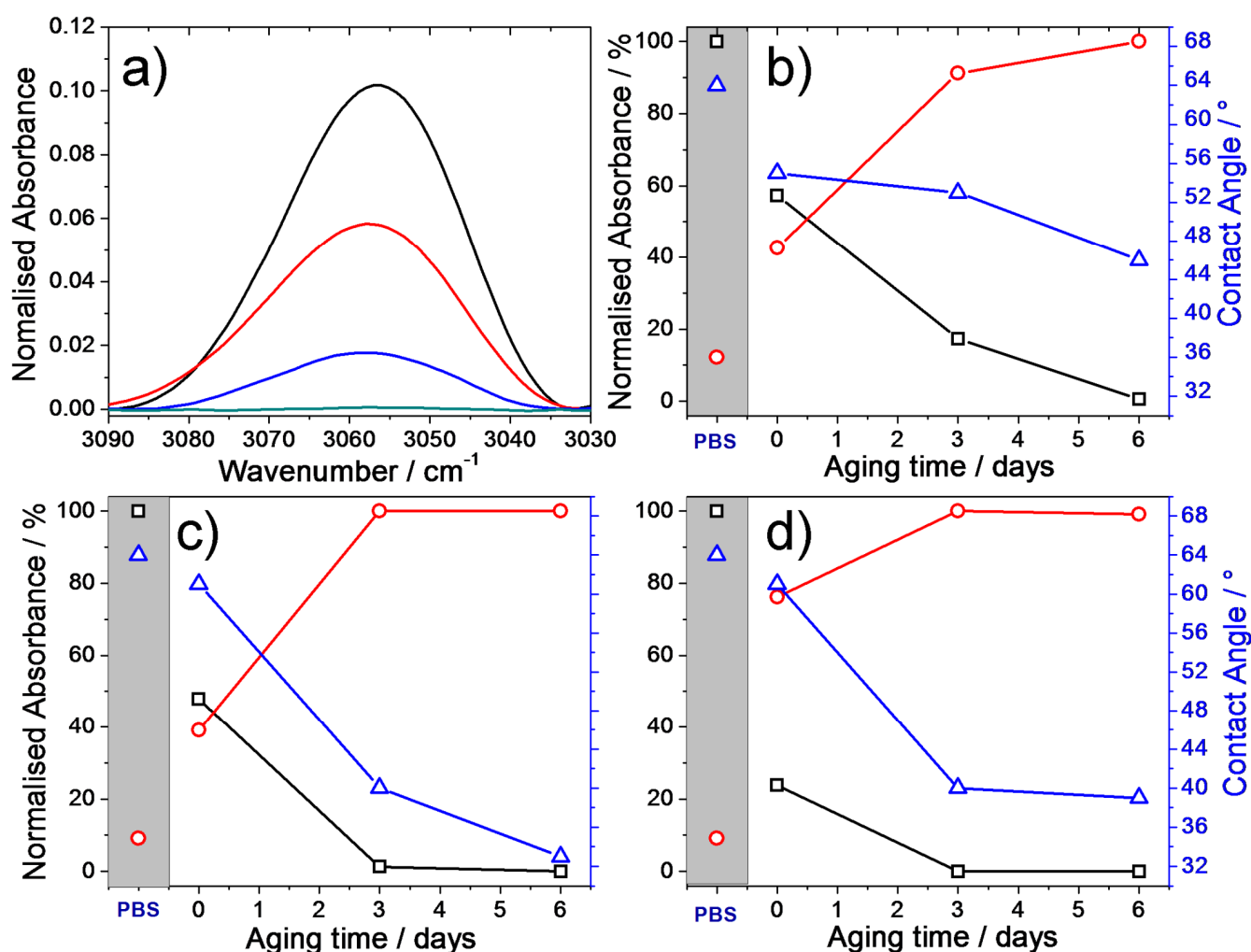


Figure 2. a) FTIR spectra of the epoxide band in a hybrid films prepared with sols at pH 14 and aged for different times (0d-red line; 3d-blue line; 6d-cyan line). The black line represents the epoxide band of a GPTMS film prepared with PBS and aged 0 day taken as a reference of closed epoxide. Normalised absorbances of epoxide band at 3057 cm⁻¹ (black squares), hydroxylated species at 3400 cm⁻¹ (red dots) and contact angles (blue triangles) as a function of aging time at different pH: b) 14, c) 7.5 and d) 3.5).

The hydrophobicity of the samples was evaluated with a Dataphysics OCA 20 by measuring the contact angle of 5 μ l water droplets deposited on the hybrid films coated on silicon substrates. The contact angle was taken as a median of at least 3 measurements.

A Wollam- α spectroscopic ellipsometer with fixed angle geometry was used for thickness measurements of films deposited on silicon substrates. The thickness was estimated by fitting the experimental data with a B-spline model for absorbing films deposited on Si substrates. The fit showed an average mean square error (MSE) of 20.

Results and discussion

The present work focuses on the design of different protocols that have been developed following specific requirements: the precursor sol should be almost solventless, the sol should allow the deposition of highly transparent coatings and also a fine tuning of the surface composition, in terms of amount of epoxy- and hydroxy-groups.

The pH control in GPTMS sols plays a key role for tuning the kinetics of both inorganic and organic reactions taking place during the sol to gel process.¹⁰ At very high pH the alkoxide species are quickly hydrolyzed, however the inorganic condensation is hampered, as well as the epoxide opening.¹¹ This allows controlling the sol reactions enabling the tuning of the surface chemistry.

As reported in our previous work,¹⁸ the deposition of highly basic GPTMS sols has proved to be a reliable protocol for the preparation of transparent crystalline films, provided that the relative humidity during deposition is kept lower than 50%. However, during this work, several attempts have been made to prepare sols with constant GPTMS/water ratio but lower pH (from almost neutral down to highly acidic conditions \approx pH 0) and they have given rise to unstable sols, that easily formed precipitates in solution or not allowed obtaining homogeneous spin-coated films due to dewetting phenomena. These limitations have been overcome observing that, when the pH of highly basic GPTMS sols is adjusted over a wide range after 12 hours from its preparation, the solution remains transparent and it allows depositing transparent films.

Moreover, we have also explored a further route to obtain a very stable sol at almost neutral pH (7.4) by adding a commercial aqueous buffer instead of a basic water solution. The changes in the hybrid film structure have been characterized by FTIR spectroscopy as a function of two parameters; aging time and pH of the precursor sols. **Figure 1** shows the absorption spectra in the 3800 - 2600 cm^{-1} range that have been normalized using the bands in the CH_2 stretching region (3100–2800 cm^{-1}) as a reference. These bands peak at 2937 and 2873 cm^{-1} and are assigned to CH_2 anti-symmetric and CH_2 symmetric stretching, respectively.^{12,13} The absorption peaks due to epoxides in GPTMS overlap with the intense bands of CH_2 stretching; the stretching modes of terminal CH_2 groups of the epoxy moiety in GPTMS¹³ are detected at 3057 and 3000 cm^{-1} and a third one at 2760 cm^{-1} . At higher

wavenumbers, around 3400 cm^{-1} , a wide band due O–H stretching is observed; this band is formed by the overlapping of several species, which can be identified as silanols and absorbed water (3200 cm^{-1}).¹⁴

The samples deposited from the sol prepared by adding PBS buffer to GPTMS, show a peculiar property; the epoxides do not open and the sol is very stable with time. No differences have been observed between 0 and 6 day aged samples (**Figure 1d** and **Table S1-S2** in ESI) and the content of water and hydroxyls is also small suggesting a low degree of hydrolysis of the sol precursors. For this reason we have deemed to take the epoxide band in the film prepared with PBS at 0 days aging as a reliable reference for estimating the percentages of epoxy ring opening in the other samples.

The spectra in **Figure 1** show that aging time and pH of the precursor sol both affect the structure of the material; in the samples prepared at pH 14, the epoxides react slowly and longer aging times are necessary to get a full opening. After 6 aging days, the epoxy ring signals disappear, indicating the formation of hydrolyzed species, glycol and polymers, poly/oligo(ethylene oxide).^{11,15} The spectra of the films at 0 day aging (**Figure 1a**) show that lowering the pH of the sols greatly affects the degree of epoxy-ring opening without aging the samples. In fact, the film prepared at pH 14 has still 58% of closed epoxide (**Figure 2b**) when compared to the film prepared with PBS at 0 day aging that was taken as a reference of 100% closed epoxides (**Figure 2a**). On the other hand, in the samples adjusted at pH 7.5 (**Figure 2c**) and 3.5 (**Figure 2d**) respectively, only 48% and 24% of the epoxides are still closed.

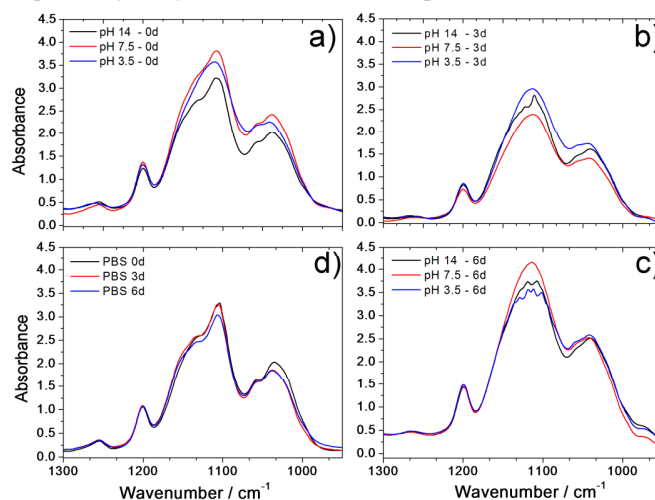


Figure 3. FTIR absorption spectra in the 1300 - 950 cm^{-1} range of the films prepared at different pH conditions (black line for pH 14, red line for pH 7.5 and blue line for pH 3.5) of the precursor sol and aging time, 0 (a), 3 (b) and 6 days (c). The spectra of the films prepared by adding the buffer at different aging times are shown in (d), (black line for aging 0 days, red line for aging 3 days and blue line for aging 6 days).

The analysis of the samples prepared at pH 14 as a function of the aging time (**Figure 2b** and **Figure S1** in the ESI) shows that after three days, the sample at pH 14 has still 18% of closed epoxide whilst at 6 aging days the epoxides disappear in all the hybrid films.¹⁶ A relevant consequence of the epoxy-ring

opening is the increase in hydroxyl species and water absorption, which has been confirmed by detailed studies in our previous works.¹⁷⁻¹⁹ The O–H stretching band increases in intensity with aging time according to the decrease in the epoxy content (**Figure 2b-2d**).

Interestingly, the property of our GPTMS system appears to be in contrast with previous findings. In particular, Gabrielli and co-workers have found that "under basic condition the precipitation process is dominant factor and opening of the epoxide ring does not occur".¹⁰ However, this conclusion has been achieved performing the study of a "water solution of GPMTS" where the GPTMS/water volume ratio is equal to 0.23; on the other hand our system is based on a "GPTMS solution of water" where the GPTMS/water volume ratio is equal to 2.25. This is a clear indication that the ratio between the two reagents plays a crucial role, especially at extreme pH, in the control of the chemical reactivity.

Besides the opening of the epoxide, the acidity of the sol also controls the condensation of the inorganic network; **Figure 3 (a-d)** shows the FTIR spectra in the 1300 - 950 cm^{-1} range. The spectra are characterized by the epoxy ring-breathing mode at 1255 cm^{-1} ,²⁰ the CH_2 wagging of propyl chain²¹ at 1200 cm^{-1} and the intense absorption band of the Si-O-Si symmetric stretching mode ($\sim 1100 \text{ cm}^{-1}$).¹⁴ This last band, in particular, shows the presence of several other overlapped bands of different intensities; the band around 1140 cm^{-1} is assigned to methoxy-groups in unreacted alkoxides and it is therefore an indication that also hydrolysis has not reached completion. However, these unreacted alkoxides are most likely inside the matrix and therefore do not participate directly to the surface chemistry of the films.

This band is clearly observed in the not-aged sample at pH 14 (**Figure 3a**) and in the buffered samples (**Figure 3d**), whilst in the other samples the silica stretching band does not show any overlapped band in the same range. At lower wavenumbers in all the samples two overlapped absorption bands at 1036 and 1046 cm^{-1} are observed; they are assigned to silica cyclic species and cages which form at highly basic conditions of synthesis, as it has been well reported in literature.¹⁷

Cyclization of organically modified alkoxides is quite commonly observed in highly acidic or basic conditions.^{22,23}

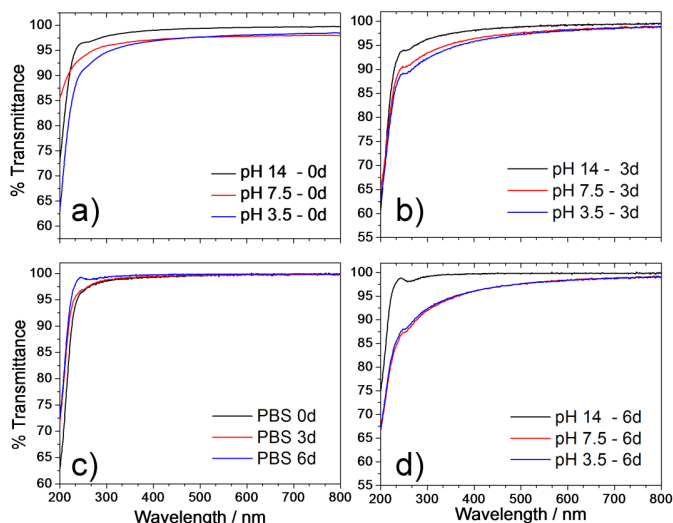
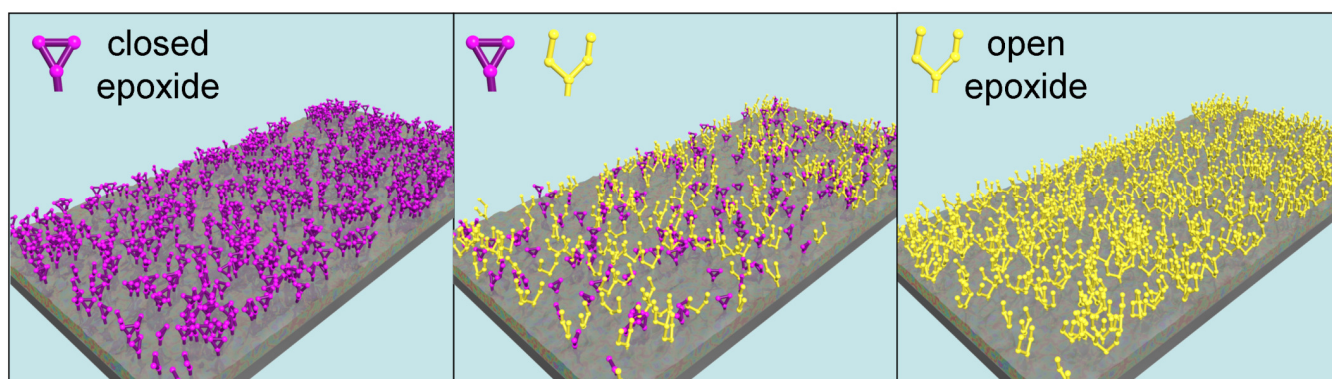


Figure 4. UV-Vis transmission spectra of the films prepared at different pH conditions (black line for pH 14, red line for pH 7.5 and blue line for pH 3.5) of the precursor sol and aging time, 0 (a), 3 (b) and 6 days (c). The spectra of the films prepared by adding the buffer at different aging times are shown in (d), (black line for aging 0 days, red line for aging 3 days and blue line for aging 6 days).

The epoxy bands, which, within this wavenumber range, do not overlap to any other signal, well support the previous discussion about the reactivity of the epoxy group: they open with aging time but do not react in the buffered GPTMS sol. On the other hand, silica condensation proceeds simultaneously with epoxy opening reactions and goes almost to completion after 6 days (**Figure 3c**); the cyclic species also reduce with time with the silica network condensation.

The optical transparency of the hybrid films has been tested by UV-vis spectroscopy; the transmittance spectra of the different samples are shown in **Figure 4**.



Scheme 2. Schematic drawing of the possible surface states: a hydroxy-covered surface with closed epoxides (a), an intermediate state with coexistence of hydroxyls groups, closed and open epoxides (b) and a surface covered by hydroxyls groups and open epoxides (c).

Transmittance %			
Samples	0 day	3 days	6 days
pH 14	99	98	99
pH 7.5	90	96	95
pH 3.5	96	95	95

Table 1. Transmittance at 365 nm measured on different films as a function of pH and aging time.

At all aging times, the different sols allow for the deposition of transparent films; despite the considerable thickness ($\approx 2 \mu\text{m}$ as estimated by spectroscopic ellipsometry) the transmittance remains around the 100-95% of the visible range for all the samples.

We have reported the changes of transmittance value at 365 nm as a function of pH and aging time of the sol; the results are shown in **Table 1**.

The data show that higher aging times and higher pH give films with higher transparency; this is expected because of the higher condensation of the structure with increasing of these parameters. The synthesis of GPTMS at different pH conditions allows obtaining a "palette" of controlled surfaces with different properties. **Scheme 2** shows a drawing of the possible surface states, which could be changed from a hydroxy-covered surface with closed epoxides (**a**), to an intermediate state with coexistence of hydroxyls groups, closed and open epoxides (**b**) and finally a surface covered by hydroxyls groups and open epoxides (**c**). The consistency of this model is supported not only by FTIR data, but also by a direct measure of the surface contact angle. The data reported in **Table 2** show that the surface becomes more hydrophilic by increasing aging time and lowering pH; the PBS samples whose surface is covered by epoxides (**Figure 1d**) is the most hydrophobic with a contact angle around 65° .

Contact Angle / $^\circ$			
Samples	0 day	3 days	6 days
pH 14	55	53	46
pH 7.5	61	40	33
pH 3.5	61	40	39

Table 2. Contact angle measured on different films as a function of pH and aging time.

Conclusions

The pH adjustment of a GPTMS sol prepared under highly basic conditions has allowed depositing hybrid organic-inorganic films of high optical transparency and controlled

hydrophilicity. Tailoring of sol aging and pH enables tuning the film surface in terms of relative amounts of epoxides and silanols. There is an interplay between epoxide opening and hydroxyl formation as a function of the aging time of the sol, which is reflected in the change of hydrophobicity of the films at a fixed pH. In general, longer aging times, which means higher condensation of silica network and opening of the epoxide, produce a decrease of contact angles. On the other hand we have also demonstrated that it is also possible to fabricate hybrid films, whose epoxides remain mostly unreacted, by adding a commercial PBS buffer to GPTMS. This protocol will thus enable the possibility of selectively coupling the epoxides on the surface with specific functionalities giving rise to an additional degree of tailoring.

The possibility of tuning the interface of the hybrid film should allow a fine control of particles binding and organic molecules coupling.

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Electronic Supplementary Information (ESI) available: absorbances of the epoxide band at 3057 cm^{-1} , normalised on the absorbance of the CH_2 stretching at 2937 cm^{-1} (Table S1); absorbances of the epoxide band at 3400 cm^{-1} , normalised on the absorbance of the CH_2 stretching at 2937 cm^{-1} (Table S2); absorbances of the epoxide band at 3057 cm^{-1} , normalised on the absorbance of the CH_2 stretching at 2937 cm^{-1} as a function of the aging time and pH (Figure S1).

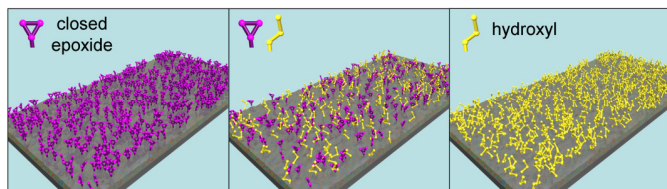
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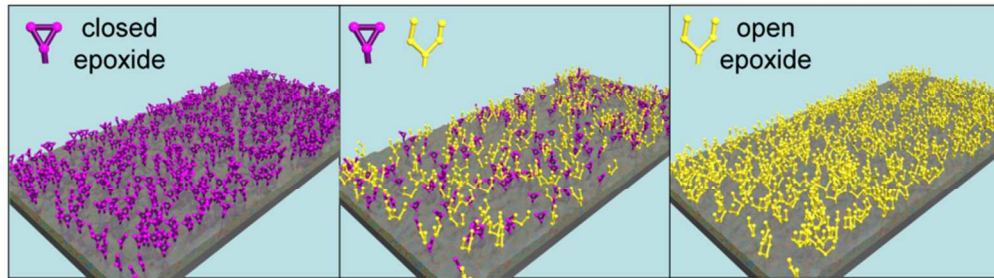
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TOC



The tuning of epoxide opening in GPTMS films allows for a smart tailoring of the surface chemistry.



80x22mm (300 x 300 DPI)