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UV/Vis Spectroscopic Properties of *N*-(2′-Hydroxy-4′-*N*,*N*-dimethylaminobenzylidene)-4-nitroaniline in Various Solvents and Solid Environments

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Summary. *N*-(2′-Hydroxy-4′-*N*,*N*-dimethylaminobenzylidene)-4-nitroaniline [*HDBN*] has been used as a model for investigating intra- and intermolecular D–A (donor–acceptor) interactions in various environments by means of UV/Vis spectroscopy. UV/Vis spectra of *HDBN* have been measured in various solvents, ethanolic solutions of different *pH*, adsorbed on silica, and in the solid state. A bathochromic shift of $\nu_{\max}$ is observed with increasing the dipolarity/polarizability and HBD (hydrogen bond donor) capacity of the solvent, which is described by means of a multiple LSE (linear solvation energy) relationship in terms of the empirical *Kamlet-Taft* solvent polarity parameters. The adsorption of *HDBN* on Aerosil® 300-silica particles in non-HBA (hydrogen bond acceptor) solvents is explained in the same sense. Mobile protons and sol–gel entrapping cause a hypsochromic shift due to protonation of the lone electron pair of the 4′-*N*,*N*-dimethylamino group. Hydroxide ions attack the 2′-hydroxy group which causes a bathochromic shift. A strong intramolecular hydrogen bond between the 2′-hydroxyl hydrogen and the imine nitrogen atom is present in the solid-state structure causing an unprecedented bathochromic shift.

Keywords. LSE correlations; Long-wavelength UV/Vis absorption band shift; Solvatochromism; Salicylidene-anilines; Intramolecular proton transfer; X-ray crystal structure analysis.

Introduction

The color change of a compound induced by external influences, *e.g.* by solvents (solvatochromism), applied stress (mechanochromism), salts (halochromisms) and/or temperature (thermochromism) has been studied intensively over the last

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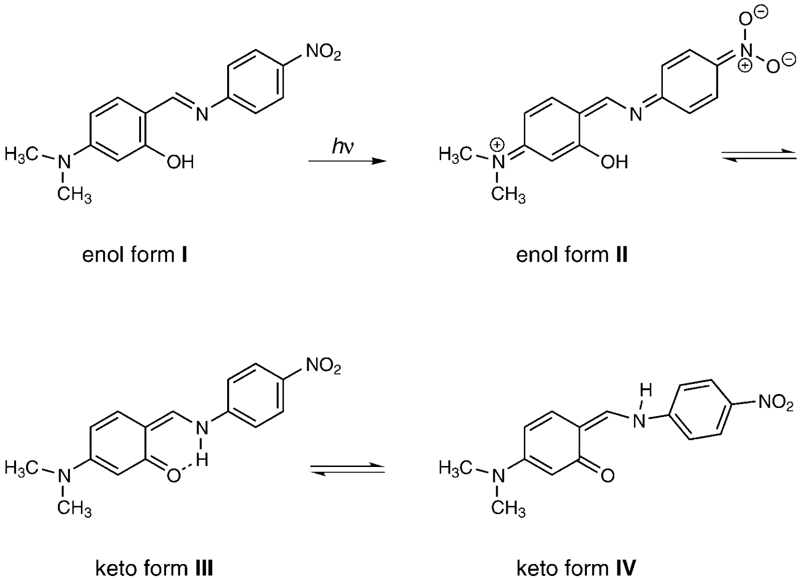
decades [1–12]. Polarity and acid–base properties of an environment are related features, but can cause quite different results of a chemical process. Whether a protonation or hydrogen bond formation without proton transfer occurs depends on many factors. Often, only one kind of the protonated or deprotonated species derived from a polar indicator shows additional solvatochromic properties [1, 13–16]. Knowledge on altering of solvatochromic properties of indicators as function of acidic strength is of importance, because those kind of probes have been widespreadly applied to investigate internal and external polarities of surfaces of macromolecular and related materials [17–26]. Push-pull substituted aromatic azomethine compounds like salicylidene-anilines (anils) can manifoldly interact with acids, bases, polar solvent molecules [27–31], and zeolites [32], because both basic and acidic sites and a dipolar delocalized π-electron system are present.

For this study, we chose the azomethine dye *N*-(2′-hydroxy-4′-*N*,*N*-dimethylaminobenzylidene)-4-nitroaniline (*HDBN*), because various intra- and intermolecular interactions with dipolar solvents, acids, and bases are possible. Each specific interaction attributes to a characteristic change in the UV/Vis spectrum (Scheme 1).

The objective is to investigate environmental influences on UV/Vis spectral changes of *HDBN* to check its suitability as a probe for material science. The well-established *Kamlet-Taft* LSE relationship has been used to study the solvatochromism [1, 33]. The simplified *Kamlet-Taft* equation applied to single solvatochromic shifts, $\mathrm{XYZ} = 1/\lambda_{\max} = \nu_{\max}$ (probe) [1, 33(b–d)], is given in Eq. (1).

$$\mathrm{XYZ} = (\mathrm{XYZ})_0 + a\alpha + b\beta + s(\pi^* + d\delta) \qquad (1)$$

$(\mathrm{XYZ})_0$ is the solute property of a reference system, for example, a non-polar medium, α describes the HBD (hydrogen bond donor) ability or acidity, β the HBA (hydrogen bond acceptor) ability or basicity, and π^* the dipolarity/polarizability of the respective solvent. δ is a polarizability correction term, which is 1.0 for



Scheme 1. Mechanism of intramolecular proton transfer in *HDBN*

aromatics, 0.5 for polyhalogenated, and zero for aliphatic solvents; *a*, *b*, *s*, and *d* are solvent-independent correlation coefficients [1, 33b].

Furthermore, UV/Vis studies of *HBDN* as function of *pH*, when adsorbed on silica, encapsulated in an ormosil glass, or as crystal powder, have been carried out in order to study the influence of qualitatively different environments.

Results and Discussion

Solent Effects on the UV/Vis Absorption Spectra and Multiple Correlation Analysis

UV/Vis absorption maxima of *HDBN* in 33 solvents and the corresponding empirical *Kamlet-Taft* solvent parameters are given in Table 1.

In increasing the solvent polarity from cyclohexane (*CH*) to formamide (Table 1), a bathochromic shift of the long-wavelength band for *HDBN* is found.

The LSEr according to Eq. (1) for *HDBN* has been calculated from the data of Table 1; $\nu_{\max}$ is expressed in cm^{-1}; *r*: correlation coefficient, SD: standard deviation, *n*: number of solvents, F: significance.

$$\nu_{\max} \cdot 10^{-3}[HDBN] = 24.661 - 1.877\pi^* - 0.715\alpha + 0.339\beta$$
$$n = 33; \quad r = 0.944; \quad \mathrm{SD} = 0.215; \quad \mathrm{F} < 0.0001 \tag{2}$$

The negative sign of *s* indicates that with increasing the solvents dipolarity/polarizability (π^*) a bathochromic shift of $\nu_{\max}$ takes place. This result demonstrates that the excited singlet state of *HDBN* becomes more stabilized when the solvent polarity increases. The negative sign of coefficient *a* indicates the formation of solute–solvent hydrogen bonds at the nitro group or azomethine moiety. However, the contribution of the α term on the UV/Vis shift of $\nu_{\max}$ [*HDBN*] is not so strong, because the coefficient *a* (≈ 0.7) is significantly smaller than coefficient *s* (≈ 1.9) [Eq. (2)]. Thus, a significant linear correlation results between $\nu_{\max}$ [*HDBN*] and solely π^* [Eq. (3)].

$$\nu_{\max} \cdot 10^{-3}[HDBN] = 24.548 - 1.867\pi^*$$
$$n = 33; \quad r = 0.724; \quad \mathrm{SD} = 0.434; \quad \mathrm{F} < 0.0001 \tag{3}$$

The β term can be neglected as shown by Eq. (4) compared to Eq. (2).

$$\nu_{\max} \cdot 10^{-3}[HDBN] = 24.805 - 1.885\pi^* - 0.693\alpha$$
$$n = 33; \quad r = 0.929; \quad \mathrm{SD} = 0.236; \quad \mathrm{F} < 0.0001 \tag{4}$$

The Effect of pH on the UV/Vis Spectroscopic Behavior of HDBN in Ethanolic Solutions

UV/Vis absorption spectra of *HDBN* in ethanolic solutions with different *pH* values are shown in Figure 1.

At $pH = 11.75$, the UV/Vis absorption maximum of *HDBN* is observed at $\lambda = 444\,\mathrm{nm}$ (bathochromic effect), whereas in acidic ethanol solution ($pH = 2.5$) this maximum appears at $\lambda = 344\,\mathrm{nm}$ (hypsochromic effect). This result is opposite

Table 1. UV/Vis absorption spectra of *HDBN* measured in diverse solvents and the empirical *Kamlet-Taft* parameters of the respective solvents from Ref. [13]

Solvent	$\nu_{\max}/10^{-3}\,\mathrm{cm}^{-1}$	α	β	π^*
cyclohexane	24.57	0.00	0.00	0.00
triethylamine	24.57	0.00	0.71	0.14
diethyl ether	24.27	0.00	0.47	0.27
tetrachloromethane	24.21	0.00	0.10	0.28
1,4-dioxane	23.98	0.00	0.37	0.55
ethyl acetate	23.92	0.00	0.45	0.55
p-xylene	23.75	0.00	0.12	0.43
1,2-dimethoxyethane	23.75	0.00	0.41	0.53
benzene	23.70	0.00	0.10	0.59
toluene	23.70	0.00	0.11	0.54
acetone	23.70	0.08	0.43	0.71
tetrahydrofuran	23.64	0.00	0.55	0.58
acetonitrile	23.58	0.19	0.40	0.75
ethanol	23.58	0.86	0.75	0.54
1-hexanol	23.58	0.80	0.84	0.40
1-decanol	23.58	0.70	0.82	0.45
1-octanol	23.53	0.77	0.81	0.40
1-propanol	23.42	0.84	0.90	0.52
methanol	23.42	0.98	0.66	0.60
chloroform	23.36	0.20	0.10	0.58
dichloromethane	23.36	0.13	0.10	0.82
pyridine	23.31	0.00	0.64	0.87
1-butanol	23.31	0.84	0.84	0.47
dimethylsulfoxide	23.26	0.00	0.76	1.00
N,*N*-dimethylacetamide	23.15	0.00	0.76	0.88
N,*N*-dimethylformamide	23.09	0.00	0.69	0.88
1,2-dichloroethane	22.99	0.00	0.10	0.81
benzonitrile	22.99	0.00	0.37	0.90
1,1,2,2-tetrachloroethane	22.68	0.00	0.00	0.95
2,2,2-trifluoroethanol	22.37	1.51	0.00	0.73
acetic acid	22.32	1.12	0.45	0.64
1,1,1,3,3,3-hexafluoro-2-propanol	22.12	1.96	0.00	0.65
formamide	22.03	0.71	0.48	0.97

to that obtained in well behaving regular solvents, where *HBD* environments caused a bathochromic UV/Vis shift. The hypsochromic shift of *HDBN* in acidic ethanol/water solutions is caused by protonation of the *N*,*N*-dimethylamino nitrogen atom. This effect strongly decreases the extent of the π-conjugated system. UV/Vis absorption spectra of *HDBN* in ethanolic solution at $pH = 5.2$ show two long-wavelength UV/Vis absorption bands, one with larger intensity and higher energy ($\lambda_{\max} = 344\,\mathrm{nm}$) and the other one with a lower intensity and bathochromically shifted ($\lambda_{\max} = 424\,\mathrm{nm}$). The position of the first UV/Vis absorption band is attributed to the protonated form of *HDBN*. The second UV/Vis band appears *ca.* $\Delta\lambda = 80\,\mathrm{nm}$ red-shifted. It is likely attributed to the presence of the quinoid

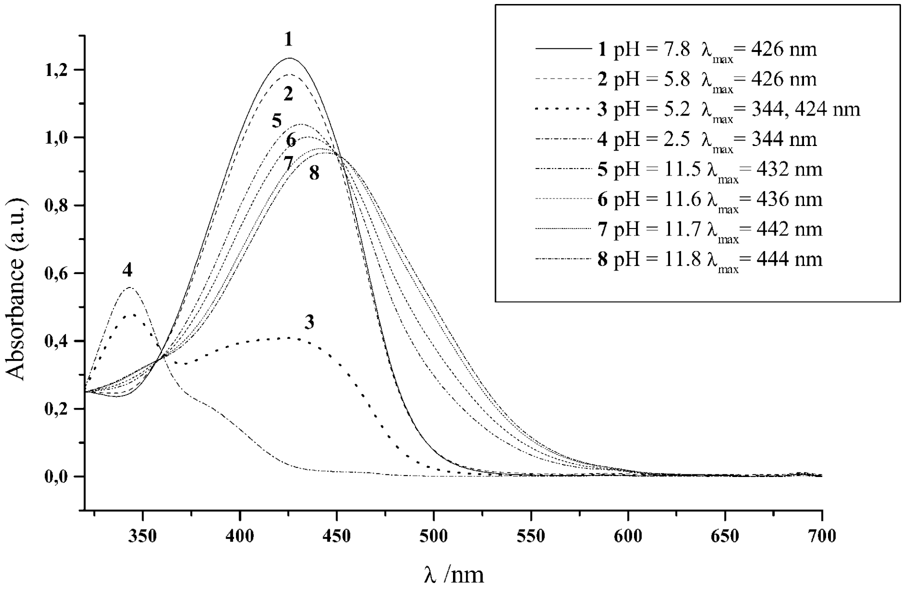


Fig. 1. UV/Vis absorption spectra of *HDBN* in ethanol at different *pH* adjusted with either aqueous HCl or NaOH

structure of *HDBN* (Scheme 1), which seems of importance in ethanolic solutions with $pH \geq 5.8$.

Adsorption of HDBN on Aerosil® 300 in Several Solvents

HDBN adsorbs readily on silica particles from non-HBA solvents such as 1,2-dichloroethane (*DCE*), 1,1,2,2-tetrachloroethane (*TCE*), toluene or benzene. This process is associated with a significant bathochromic shift of the solvatochromic UV/Vis absorption band. Shape of the UV/Vis spectra are not changed by adsorption (Table 2).

The increased stabilization of the polar ionic structure (enol form II of Scheme 1) on silica is in agreement with the result obtained in the strong dipolar solvents 2,2,2-trifluoroethanol, 1,1,1,3,3,3-hexafluoro-2-propanol, or formamide.

Measured $\nu_{\max}$ of *HDBN* adsorbed on Aerosil® are in good agreement with calculated $\nu_{\max}$ of *HDBN* on Aerosil® in several solvents (using Eq. (5) and α and π^* of Aerosil® solvent from Ref. [11b], Table 2, values in parenthesis). Thus, the silica surface behaves like HBD solvents. Hydrogen bond formation, which may occur solely at the oxygen atoms of the nitro group when *HDBN* is adsorbed on silica, enhances the electron push–pull conjugation effect. Hydrogen bond formation at the dimethylamino and/or hydroxyl groups of *HDBN* adsorbed on silica play no role. According to results from the literature, interaction of silanol groups at the nitro group of push–pull aromates are preferred rather than at *N*,*N*-dimethylamino or OH groups [26]. Moderately strong HBA solvents ($\beta > 0.3$), such as triethylamine, diethylether, tetrahydrofuran, dimethylformamide, and acetonitrile, themselves interact strongly with silanol groups of silica particles [34]. Therefore, *HDBN* does not completely adsorb onto silica from those solvents and sometimes

Table 2. UV/Vis absorption maxima of *HDBN* when adsorbed onto dried Aerosil® 300 in various solvents (suspension) and the difference of the wave number ($\Delta\nu_{\max}$) to $\nu_{\max}$ [*HDBN*] in the pure solvent as well as the comparison with expected $\nu_{\max}$ values from the LSEr Eq. (5) using independently determined α and π^* values of Aerosil®/solvent compositions

Solvent	$\nu_{\max} \cdot 10^{-3}\,\mathrm{cm}^{-1}$	$\Delta\nu_{\max} \cdot 10^{-3}\,\mathrm{cm}^{-1}$
toluene ($\alpha = 1.14; \pi^* = 0.97$)[a]	21.65 (22.24)[b]	2.05
CCl_4 ($\alpha = 1.52; \pi^* = 0.56$)[a]	22.32 (22.7)[b]	1.89
benzene	21.83	1.87
p-xylene	22.08	1.67
DCE ($\alpha = 1.15; \pi^* = 1.01$)[a]	21.74 (21.17)[b]	1.25
CH_2Cl_2 ($\alpha = 0.98; \pi^* = 1.14$)[a]	22.27 (22.07)[b]	1.09
1,1,2,2-tetrachloroethane	21.60	1.08
$CHCl_3$ ($\alpha = 0.95; \pi^* = 1.1$)[a]	22.37 (22.14)[b]	0.99
dimethylsulfoxide	22.62	0.64
acetonitrile[c]	23.70	− 0.12
diethyl ether[c]	24.15	0.12
dimethylformamide[c]	23.15	− 0.06
tetrahydrofuran[c]	23.58	0.06
triethylamine[c]	24.57	0.00

[a]The parameters in parenthesis are those for the Aerosil®/solvent interface, independently determined with Fe(*phen*)$_2$(CN)$_2$ and *Michler*'s ketone as surface polarity indicator from Ref. [11]; [b]the value in parenthesis is the calculated $\nu_{\max}$ value for adsorbed *HDBN* from Eq. (5), using the independently determined α and π^* values from Ref. [11b]; [c]*HDBN* is only partly adsorbed

no UV/Vis shift was observed for the solvent/Aerosil® system compared to the pure solvent (Table 2). The polarity difference of the silica/solvent interface compared to the pure solvent is the largest for solvents with high dipolarity/polarizability, π^* value, of the solvent.

UV/Vis Spectroscopic Results of HDBN in the Solid-State

A crystal powder of *HDBN* and samples entrapped in a special sol–gel glass with a polarity comparable to that of methanol were used for these experiments.

X-ray crystal structure analysis of *HDBN* was carried out in order to interpret the intense red color of the crystals in relation to structural features. *HDBN* can be crystallized from benzene at 25°C as red blocks, monoclinic space group P2$_1$/c, with $a = 1683.38(10)$, $b = 723.06(2)$, $c = 1159.95(2)$ pm, $\beta = 109.568(2)°$, $V = 1330.33(4) * 10^6\,\mathrm{pm}^3$ and $Z = 4$. Selected interatomic bond distances (pm) and angles (°) are listed in the legend of Fig. 2. Crystal data are deposited in Cambridge data centre.

Relevant interatomic bond lengths of O3–C9, C7–C8, C8–C9, and N1–C7 are 135.27(19), 143.18(17), 142.2(2) and 129.9(2) pm (Fig. 2). The distances of O3–C9 and C7–C8, compared to typical C=O (122.2 pm) and C=C bonds (134.0 pm), indicate significantly elongated conjugated enones [35].

These results suggest that *HDBN* exists in the solid-state in the enol form. A strong intramolecular hydrogen bond between the hydroxyl hydrogen atom and the

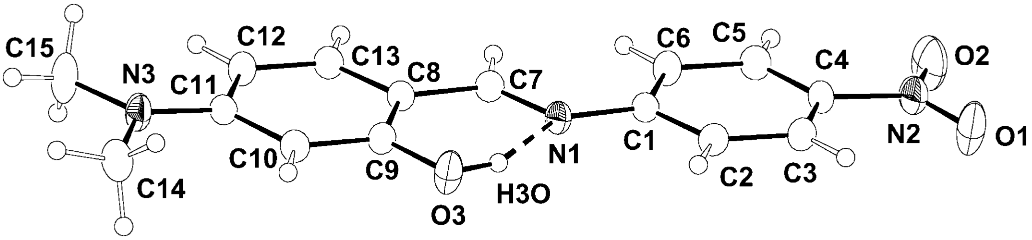


Fig. 2. ZORTEP PLOT (50% probability level) of *HDBN*. Selected bond lengths [pm]: C(9)–O(3) 135.27(19), O(3)–H(3O) 88.0(2), C(7)–C(8) 143.18(17), C(8)–C(9) 142.2(2), N(1)–C(1) 140.87(15), N(1)–C(7) 129.9(2); selected bond angels [°]: C(6)–C(1)–N(1) 124.86(14), C(7)–N(1)–C(1) 121.49(13), N(1)–C(7)–C(8) 121.69(14), C(9)–C(8)–C(7) 122.59(14), C(9)–O(3)–H(3O) 106.2(17); selected torsion angles [°]: C(7)–N(1)–C(1)–C(2) 176.54(15), N(1)–C(7)–C(8)–C(9) 1.4(2), C(7)–C(8)–C(9)–O(3) $-0.4(2)$, C(1)–N(1)–C(7)–C(8) $-179.57(13)$

imine nitrogen atom exists (Fig. 2) which holds *HDBN* in a planar conformation optimizing the molecular orbital overlap.

The diffuse UV/Vis reflectance spectrum of *HDBN* measured as fine-powder shows a broad UV/Vis absorption section with three small poorly resoluted maxima at $\lambda_{max} = 337$, 380, and 492 nm. The first two UV/Vis bands relate to contributions of NH and keto tautomeric structures, respectively, whereas the third one likely reflects an electronic transition derived from the strong dipolar oppositely arranged aggregation of dye molecules.

When *HDBN* is entrapped within a rigid ormosil host, a single UV/Vis absorption is observed at $\lambda_{max} = 355$ nm relating to the behaviour in aqueous solution at $pH = 2.5$ [39]. This result is in contrast to those when anils are mobile guests in highly polar Y-zeolite super cages, where the zwitterionic form becomes stabilized [32].

Conclusion

HDBN reflects environmental influences by manifold shifts of its absorption band in the UV/Vis spectrum. The solvent influence on ν_{max} of *HBDN*, its solvatochromism, can be quantitatively expressed by means of a LSE relationship using the empirical *Kamlet-Taft* solvents parameter set. The LSE analyses show that dipolar interactions preferably contribute to the environmentally induced color changes. Genuine solvatochromism is suppressed if mobile protons co-interact with the *N*,*N*-dimethylamino group. This is observed in acidic aqueous media and in sol–gel glasses. The intense red color of the *HBDN* crystal is attributed to intermolecular interactions of the oppositely arranged dipolar molecules in the solid-state.

The introduction of hydrophobic and hydrophilic functionalities at the periphery of salicylidene-anilines are expected to change solid-state structures in relation to UV/Vis absorption properties, which makes this kind of compound promising for investigating chromophores in terms of environmental effects relating to optical properties for application.

Experimental

Materials and Methods

The reagents were of analytical grade and purchased from Aldrich and Acros. Solvents for the solvatochromic measurements were used as commercially available in the highest available quality (analytical or spectroscopic grade) and were additionally dried and purified according to standard procedures [1a, 36, 37].

Elemental analysis was determined with a Vario-EL analysis. NMR measurements were recorded at 20°C on a Varian Gemini 300 FT NMR spectrometer, operating at 300 MHz for ^{1}H and at 75 MHz for ^{13}C. The signals of the solvents ($CDCl_3$) were used as internal standards. The UV/Vis absorption spectra of freshly prepared solutions were measured by means of MCS 400 diode-array spectrometer from Carl Zeiss Jena connected with an immersion cell (TSM 5) via glass-fiber optics. A diffuse reflectance accessory was attached to the spectrometer for diffuse reflectance measurements, which were carried out with properly characterized powdered samples using $BaSO_4$ powder as a reference.

Aerosil® 300 (particle diameter: 7 nm; specific surface area (BET): $240\,m^2g^{-1}$) was a kind gift of Degussa (Frankfurt/Main). It was heated at 400°C for 12 h before use. After cooling to 25°C under dried argon, a solution of the probe dye in the corresponding solvent (about $10^{-5}\,mol/dm^3$) was added to Aerosil® 300. Care must be taken to avoid overloading the surface with the dye used, as multi-layer adsorption is expected in solution at higher concentrations as well as interfering absorptions from the nonadsorbed dye from the solution.

Correlation Analysis

Multiple regression analysis was performed with the Origin 5.0 statistic programs.

Structure Determination

The crystal structure of *HDBN* was determined using single-crystal X-ray diffraction methods as reported in Ref. [12a–c]. The complete data collection parameters and details of the structure solution and refinement are available from the Cambridge Crystallographic Data Centre, on quoting the depository number CCDC 188074.

Synthesis of HDBN

Synthesis of *HDBN* was carried out according to Ref. [28].

Preparation of Sol–Gel Materials

The *HBDN* doped ormosil glass was synthesized from a 1:1 mixture of tetramethoxysilane (*TMOS*) and methyl trimethoxysilane (*MTMOS*), according to an established sol–gel procedure [39].

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