

# Pressure-Sensitive Conducting and Antibacterial Materials Obtained by *in Situ* Dispersion Coating of Macroporous Melamine Sponges with Polypyrrole

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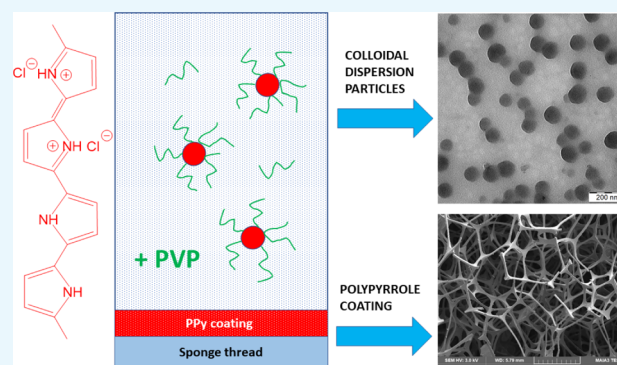
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**ABSTRACT:** Melamine sponges were coated with polypyrrole during the *in situ* polymerization of pyrrole. The precipitation polymerization was compared with the dispersion mode, that is, with the preparation in the presence of poly(*N*-vinylpyrrolidone) and nanosilica as colloidal stabilizers. The coating of sponges during the dispersion polymerization leads to the elimination of the undesired polypyrrole precipitate, improved conductivity, and increased specific surface area. The sponges were tested with respect to their conductivity and as pressure-sensitive conducting materials with antibacterial performance.



## 1. INTRODUCTION

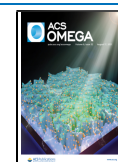
Conducting polymers,<sup>1,2</sup> such as polypyrrole,<sup>3</sup> are currently prepared by the oxidation of respective monomers in aqueous medium, iron(III) chloride being an oxidant of choice in the oxidation of pyrrole.<sup>4,5</sup> The oxidation takes place in aqueous medium, and any object in contact with the reaction mixture becomes coated with a thin polymer film. This is so-called surface polymerization.<sup>6</sup> After the interface is coated and reactants are still present in the reaction mixture, the precipitation polymerization continues in the aqueous medium and macroscopic polymer aggregates are produced and isolated as a powder.

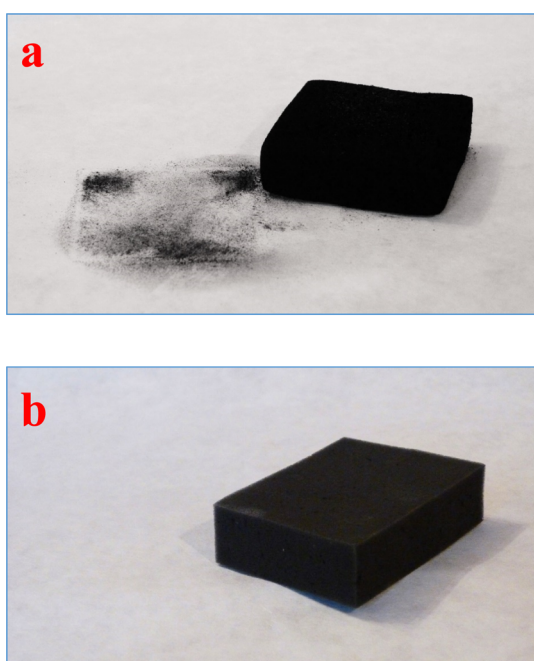
The conducting polymers alone are intractable. For this reason, their deposition on suitable materials is a solution for most applications. Polymer sponges are attractive substrates for the coating with conducting polymers due to their low weight, open-cell macroporosity, high specific surface area, chemical stability, and elasticity. This makes them good candidates for sorbents applicable in biomedicine,<sup>7</sup> analytical chemistry,<sup>8</sup> solar steam generation,<sup>9</sup> energy storage,<sup>10</sup> and especially in environmental issues, such as water-pollution treatment.<sup>11–14</sup> Melamine/formaldehyde sponges are commercially available, and they are used as flame-retardant sound-proofing and thermal insulation or as abrasive cleaners. The coating with polypyrrole endows them with additional properties, such as the conductivity,<sup>15</sup> electroactivity, and ability to sense humidity<sup>16</sup> or to adsorb organic dyes<sup>12,13</sup> and various

pollutants, including oil spills<sup>17</sup> and heavy-metal ions.<sup>18</sup> The conducting and electroactive coating enables to control the properties by applying or recording an electric potential, for example, in stimulation or monitoring of biological objects,<sup>2,19</sup> the heating of sponges by passing electric current being another example.<sup>17</sup>

When applied to the coating of macroporous melamine sponges by surface polymerization of pyrrole,<sup>15</sup> the precipitated polymer produced at the same time remains entrapped in the macroporous template. The precipitate cannot be removed even by excessive washing. For various applications, this may be inconvenient because the polymer leaves the sponge during the processing, for example, cutting or squeezing of the sponge, and contaminates the surrounding space (Figure 1a). The presence of a fine polypyrrole precipitate can be eliminated by the addition of a water-soluble polymer stabilizer, for example, poly(*N*-vinylpyrrolidone) (PVP) (Figure 1b), or nanocolloidal silica. Its presence converts a macroscopic polypyrrole precipitate to a colloidal dispersion that is easily washed out from the sponge. This approach, the coating of sponges in the

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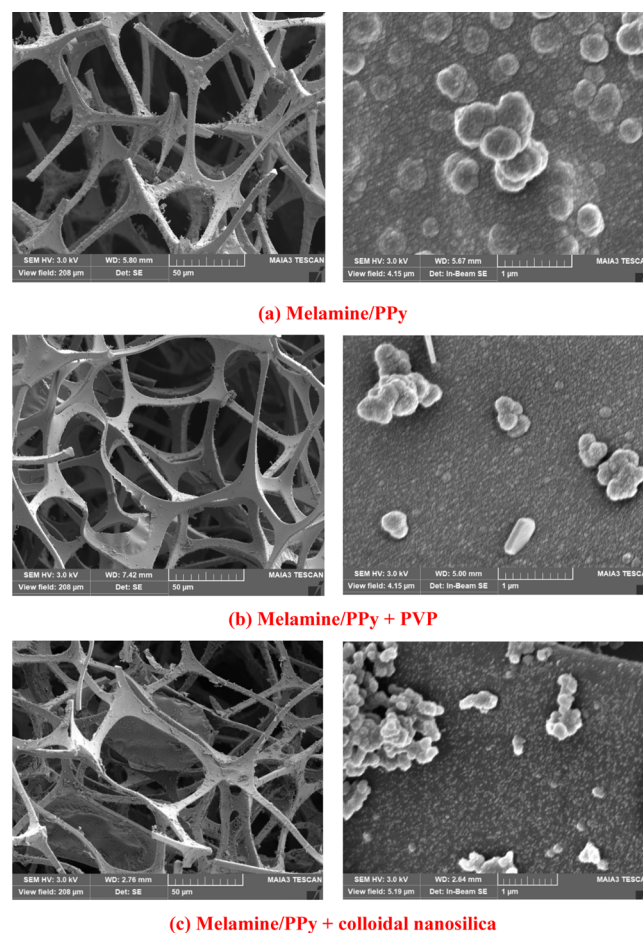
**Figure 1.** Melamine sponges after being compressed with hand and released: (a) polypyrrole coating and (b) reduced polypyrrole content deposited in the presence of PVP.

course of dispersion polymerization of pyrrole, is demonstrated in the present report.

## 2. RESULTS AND DISCUSSION

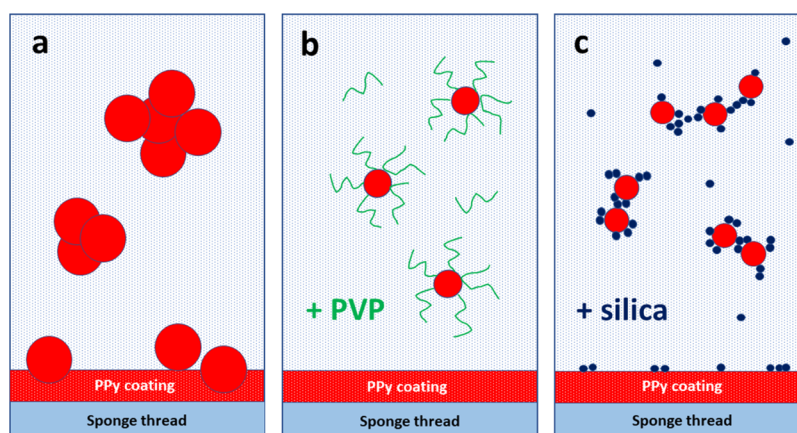
**2.1. Polypyrrole Coating.** Pyrrole is oxidized in aqueous medium to polypyrrole. Any surface in contact with this medium, here the threads of melamine sponge, becomes coated with a thin conducting polymer film<sup>20–22</sup> (Figure 2). The typical thickness of films produced in *in situ* precipitation polymerization is 100–200 nm.<sup>21</sup> In the absence of any additive, globular polypyrrole particles are produced in the medium surrounding the sponge, that is, inside the macropores, and they are also incorporated into the growing film or adhere to its surface (Figure 2a). The macroscopic precipitate is not observed within the macropores by microscopy due to the way of the sample preparation for microscopy, but the incorporation of globular polypyrrole into coating is clearly

visible (Figure 3a). The presence of the free polypyrrole is only revealed when the sponges are compressed or cut as an



**Figure 3.** Micrographs of melamine sponge coated with polypyrrole (a) in the absence of any stabilizer, (b) in the presence of PVP, or (c) colloidal nanosilica taken at lower (left) and higher magnification (right).

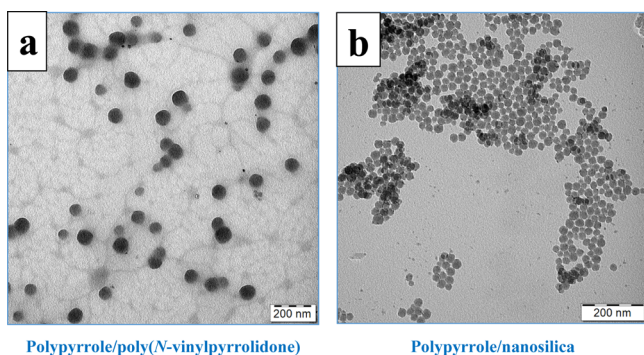
emanating “smoke” and the surrounding area gets “dirty” (Figure 1a). No changes in the melamine skeleton associated with the coating have been detected (Figure 3).



**Figure 2.** Polypyrrole coating produced on the threads of melamine sponge during (a) precipitation polymerization and in the dispersion mode stabilized (b) with PVP or (c) colloidal nanosilica.



If the same experiment is carried out instead of water in the solution of PVP, the conducting polymer is produced as colloidal dispersion stabilized with this water-soluble polymer.<sup>21,23–25</sup> (Figure 2b). The typical size of colloidal particles is about 100 nm (Figure 4a). They are easily removed from



**Figure 4.** Transmission electron micrograph of (a) polypyrrole dispersion particles stabilized with PVP and (b) aggregates produced in the presence of colloidal nanosilica.

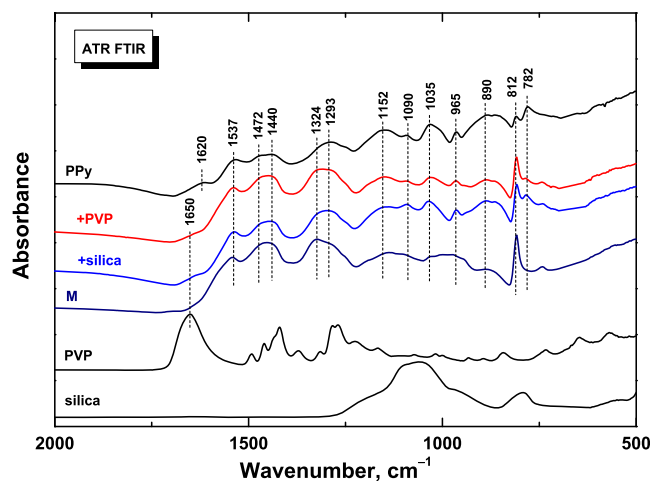
open-cell pores with the size of tens of micrometres (Figure 3b). Also, outside the sponge, a stable colloid, a “soluble” polypyrrole, is produced. Such preparation is referred to proceed in the dispersion mode. The polypyrrole films are produced on immersed substrates also in this case. Compared to the previous case, they are thinner and smoother,<sup>24</sup> with some colloidal particles occasionally attached to the film surface (Figure 3b). The analogous studies on polyaniline demonstrated that PVP does not become incorporated into the conducting polymer film.<sup>24</sup> There is no free polypyrrole precipitate which would later contaminate the surroundings during sponge processing (Figure 1b).

The water-soluble PVP can also be replaced with an inorganic stabilizer (Figure 2c), colloidal nanosilica, here with a 35 nm particle size. In the case of polyaniline, colloidal dispersions of spherical nanoparticles are produced too.<sup>26–28</sup> The presence of nanosilica prevents the formation of a macroscopic polypyrrole precipitate, but the dispersions are not been stable and sedimented (Figure 4b). Electron microscopy reveals polypyrrole/nanosilica aggregates (Figure 3c), but the underlying polypyrrole films are of good quality (Figure 3c). At closer inspection and in contrast to PVP, silica nanoparticles are uniformly incorporated onto the polypyrrole film surface.<sup>27</sup>

The content of the conducting polymer was calculated from the increase in mass after polypyrrole deposition as 21.1 wt % for the sponge prepared in the absence of additives, 6.9 wt % in the presence of PVP, and 9.2 wt % with nanosilica. In the coating during the precipitation polymerization of pyrrole, an additional part of polypyrrole is present in the macropores as free powder (Figure 1a). This is absent in the dispersion coating, and the content of polypyrrole in sponges is reduced correspondingly. It has also been demonstrated that the coatings in the presence of steric stabilizers, PVP<sup>24</sup> and nanosilica,<sup>27</sup> are thinner than those prepared in their absence. This is additional reason for the reduction in conducting polymer content. The increase in mass after the coating procedure may be, in principle, due to the incorporation of the stabilizers to the polypyrrole coating. This was proved to be absent at least in the case of PVP,<sup>24</sup> but it is present, to some

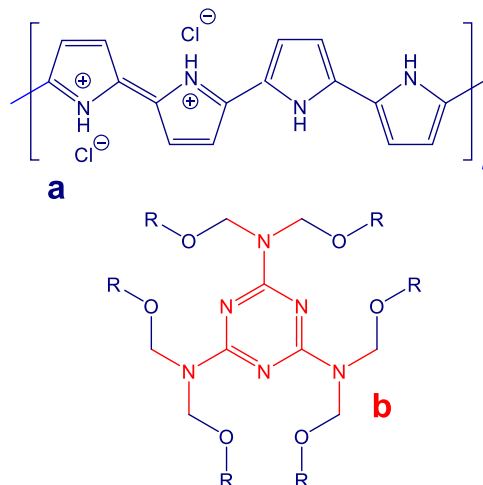
extent, in the case of nanosilica (Figure 3c);<sup>27</sup> the residue in the thermogravimetric analysis performed in air allows us to estimate the silica content in the sponge to 2 wt %.

**2.2. FTIR Spectra.** The Fourier transform infrared (FTIR) spectrum of the melamine sponge coated with polypyrrole is very close to the spectrum of PPy powder<sup>5</sup> (Figure 5). This is



**Figure 5.** ATR FTIR spectra of the melamine sponge coated with polypyrrole in the absence (PPy) and in the presence of PVP (+PVP) or colloidal nanosilica (+silica). The spectra of melamine (M), PVP, and nanosilica are shown for the comparison.

due to the similar chemical bonding in the molecular structure of both nitrogen-containing entities (Figure 6). Melamine

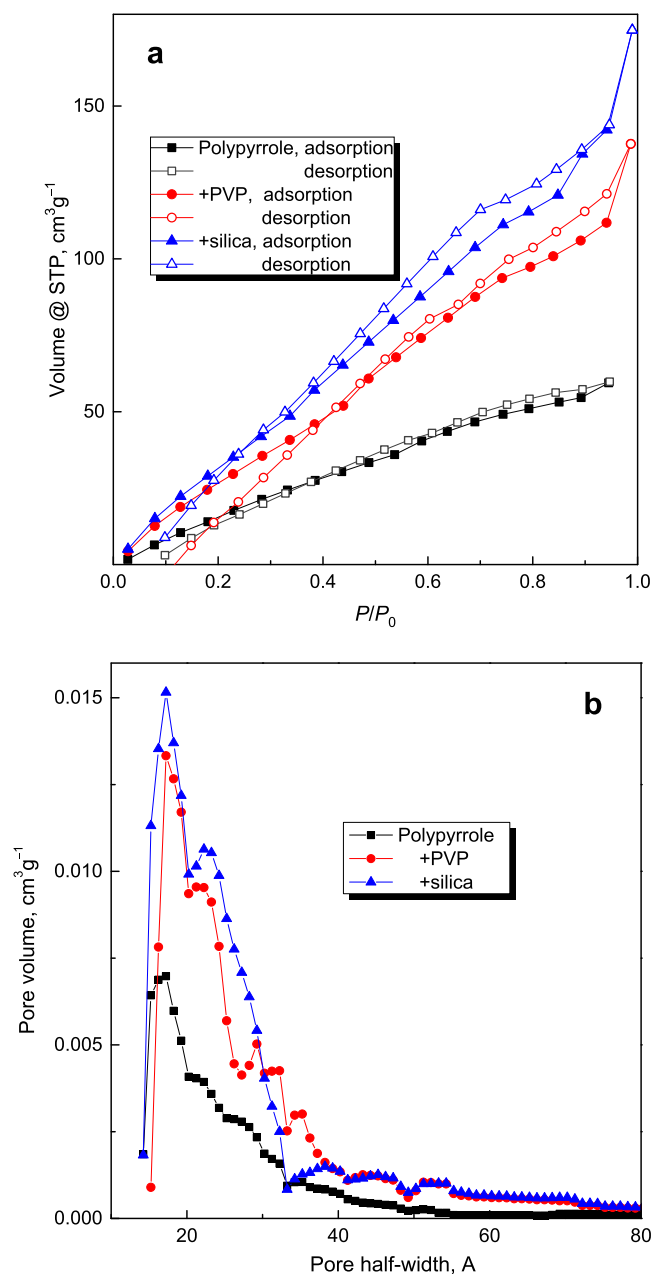


**Figure 6.** Molecular structure of (a) polypyrrole salt (hydrochloride) and (b) melamine incorporated in melamine/formaldehyde sponge. R = H or a linkage to another melamine unit.

sponge manifests itself by the characteristic peak at 812  $\text{cm}^{-1}$  (spectrum M).<sup>15</sup> This peak is present even after the coating with polypyrrole, but its contribution becomes reduced (spectra PPy, +PVP, and +silica). This reflects the fact that the coatings in the presence of the colloidal stabilizer are thinner than those in their absence<sup>24</sup> but still penetrable to the infrared radiation. The peaks of poly(*N*-vinylpyrrolidone) are absent (spectrum +PVP) because this polymer is not incorporated into polypyrrole coatings.<sup>24</sup> Silica, on the other hand, is present, but its content is low, limited to a few percent,

and silica does not display any sharp absorption peaks. For this reason, the presence of silica manifests itself only as a modest broad-band shift of a baseline.

**2.3. Surface Properties.** The specific surface area and pore volume of the sponges coated with polypyrrole in the dispersion mode are higher compared to the standard coating (Figure 7, Table 1). This is positive news for most applications.



**Figure 7.** (a) Adsorption isotherms and (b) pore size distribution of the melamine sponge coated with polypyrrole (squares) compared with the coating in the presence of PVP (circles) or colloidal nanosilica (triangles).

In the standard coating, the macropores seem to be blocked by the polypyrrole precipitate. Almost the third of sample weight was lost at degassing before and in the course of surface area determination due to the free polypyrrole particles, which are not attached to the sponge skeleton and easily leave the macroporous interior (Figure 1a). Such a mass decrease has

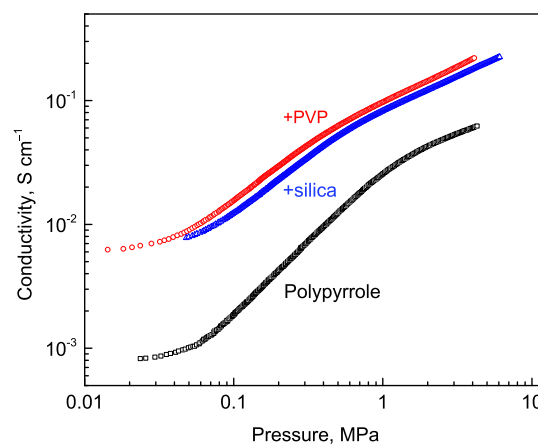
**Table 1.** Specific Surface Area and Pore Volume of Melamine Sponge Coated with Polypyrrole Compared with the Coating in the Presence of PVP or Colloidal Nanosilica

sample	$S, \text{m}^2 \text{g}^{-1}$	$V, \text{cm}^3 \text{g}^{-1}$
melamine sponge <sup>a</sup>	$39.2 \pm 0.5$	$0.071 \pm 0.001$
polypyrrole-coated sponge	$75.2 \pm 0.6$	$0.081 \pm 0.002$
+PVP	$158 \pm 5.1$	$0.233 \pm 0.015$
+silica	$172 \pm 11$	$0.241 \pm 0.012$

<sup>a</sup>Taken from ref 15.

not been observed when the coating took place in the dispersion mode (Figure 1b). Mass reduction was taken into account when evaluating the specific surface area.

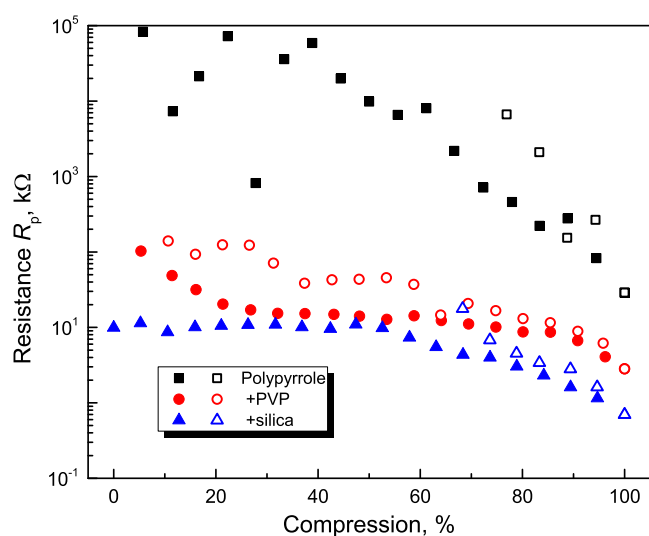
**2.4. Conductivity.** The conductivity is an intensive electrical characteristic of the material that does not depend on the size and shape of the samples. Its determination by the four-point van der Pauw method as a function of pressure is not trivial, and the reader is referred to the Experimental Section for details. The conductivity of the sponges coated with polypyrrole increases by about two orders of magnitude during the compression as expected (Figure 8) because the



**Figure 8.** Conductivity of the melamine sponges coated with polypyrrole in the absence and in the presence of PVP or colloidal nanosilica.

number of conducting pathways per infinitesimal sample cross-section increases. The sponge coated with polypyrrole in the dispersion mode in the presence of PVP or colloidal nanosilica has a comparable conductivity. After compression, the conductivity reaches the order of 10<sup>-1</sup> S cm<sup>-1</sup> and approaches the conductivity of globular polypyrrole, which is of the same order of magnitude.<sup>5</sup> The coating of sponges with polypyrrole in the precipitation mode, despite the higher polypyrrole content, is lower by about 1 order of magnitude. We can speculate that, in this case, free polypyrrole particles that are not attached to the sponge threads do not contribute to the conduction.

**2.5. Resistance.** For the practical applications, for example, in pressure sensing, the change in the sample resistance (extensive quantity), rather than the conductivity (intensive quantity), is of prime interest. In this case, the resistance of the sample decreases when the pressure is applied (Figure 9). The change in resistance is the result of the increasing conductivity (Figure 8) along with the reduced length of the cylindrical sample during compression. The results of conductivity and resistance determination cannot be directly compared. The



**Figure 9.** Resistance of the melamine sponge coated with polypyrrole in the absence (squares) and in the presence of PVP (circles) or colloidal nanosilica (triangles) during compression (full symbols) and pressure release (open symbols).

former method uses a direct current and pressures approaching 10 MPa, while the latter exploits the alternating current of 1 kHz frequency at pressures of up to  $\approx 0.1$  MPa. In the last case, a parallel resistance of the  $R_p - C_p$  equivalent electrical circuit of the sponge (circuit selected based on the inner sample shape) is evaluated along with the compression or expansion.

The resistance  $R_p$  of the sponge coated with polypyrrole in the absence of any stabilizer increases more than four orders of magnitude at full compression (Figure 9). By 100% compression, we mean the state at the maximum applied pressure 170 kPa. From the practical point of view, the use of the sponge at low compression (up to 40%) is limited as the resistance  $R_p$  is in the order of M $\Omega$ . This brings problems associated with electrical noise added to the measured signal. Hence, the sponge coated with polypyrrole only can be considered as insensitive to low pressures equal up to approx. 40% of compression. We assume that at low pressures, the free polypyrrole is distributed within the macropores, and only later, at higher pressure, it becomes squeezed between polypyrrole-coated threads and starts to participate in the conduction. There is a marked memory effect, that is, the length of the sponge does not completely recover after the pressure is released.

The sponge coated in the presence of PVP is less sensitive to the small pressure changes, and the resistance  $R_p$  decreases only by two orders of magnitude, but the resistance  $R_p$  is generally lower than in the preceding case (Figure 9). This may be of benefit in compressible and flexible supercapacitor electrodes. The memory effect is less pronounced compared with the polypyrrole coating in the absence of the stabilizer. The introductory experiment proves that the memory effect can be eliminated using pressures below  $\approx 10$  kPa. Finally, sponges coated by polypyrrole and in the presence of nanosilica exhibit the lowest resistance  $R_p$  (Figure 9). This sponge is again almost insensitive to initial compression up to approximately 40%. Moreover, a similar memory effect can be also observed here.

In conclusion, the addition of PVP to the synthesis protocol brings the benefits in the form of a moderate decrease in

resistance which allows us to use the sponge in the whole compression range along with a modest memory effect, which allows the sponge to expand back to the initial length.

**2.6. Antibacterial Properties.** Antibacterial properties are welcome for materials planned to be applied in many fields of industry and in biomedicine, and water-pollution treatment may serve as an example. The melamine-formaldehyde/poly(vinyl alcohol) composite was reported to have bactericidal effects,<sup>11</sup> and such a behavior thus might be expected from the melamine-formaldehyde/melamine-formaldehyde sponge alone. Another study on neat melamine sponges, however, has not revealed any antibacterial properties.<sup>29</sup> On the other hand, polypyrrole was found to display marked antibacterial properties,<sup>30</sup> which could be still enhanced by incorporation of silver nanoparticles.<sup>31</sup>

The results indicate that the presence of polypyrrole-coated sponges in the bacterial broth significantly reduces the number of colony-forming units (Table 2). The effect of melamine

**Table 2. Concentration of Colony-Forming Units (CFU mL<sup>-1</sup>) in Bacterial Suspensions in the Presence of Melamine Sponges Coated in Various Ways with Polypyrrole**

coating	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>
reference	$8.4 \times 10^6$	$1.3 \times 10^8$
no coating	$1.5 \times 10^6$	$1.3 \times 10^7$
PPy coating	$1.5 \times 10^5$	$3.8 \times 10^6$
+PVP	$7.5 \times 10^4$	$6.8 \times 10^5$
+silica	$3.7 \times 10^2$	$1.1 \times 10^5$

sponge alone is only marginal, but the coating with polypyrrole reduced the number of bacterial units by 3–4 orders of magnitude. Especially, the polypyrrole coating with incorporated nanosilica is efficient in antibacterial performance (Table 2), possibly due to the expected higher hydrophilicity and closer interaction with bacteria. In the present case, this can be assigned not only to the restricted bacterial growth but, alternatively or in the combination, also due to the adsorption and subsequent deactivation of bacteria on the sponge.<sup>30</sup> Regardless of the mechanism, the sponges can be considered as materials with good antibacterial performance.

### 3. CONCLUSIONS

The dispersion coating of melamine sponges with polypyrrole produced in the course of dispersion polymerization of pyrrole in the presence of PVP or colloidal nanosilica has several positive effects compared to the coating in the absence of stabilizers: (1) no contaminating polypyrrole precipitate produced inside the sponge, (2) the improved conductivity by 1 order of magnitude, (3) reduced memory effect upon compression and release, and (4) enhanced specific surface area. These polypyrrole-coated sponges can be applied as pressure-sensitive materials applicable in biomedicine with the benefit of antimicrobial performance.

### 4. EXPERIMENTAL SECTION

**4.1. Preparation.** Commercial open-cell macroporous melamine-formaldehyde sponges of the Basotect type (BASF AG, Germany) advertised as miraculous sponges were delivered in  $105 \times 62 \times 25$  mm<sup>3</sup> blocks from a local distributor Drogerie ZDE (Czech Republic). They were coated with polypyrrole in an aqueous reaction mixture containing 0.05 M pyrrole and 0.125 M iron(III) chloride hexahydrate



(Sigma-Aldrich).<sup>15</sup> For the preparation of polypyrrole in 1 L of the reaction mixture, pyrrole (3.35 g) and iron(III) chloride hexahydrate (33.8 g) were dissolved separately in water. The volume of solutions was adjusted to 0.5 L. Both solutions were precooled to 4 °C, mixed, and poured quickly over the melamine sponge. The sponge was gently squeezed to assure the penetration of the reaction mixture into the sponge. The precipitation polymerization of pyrrole was left to proceed for 1 h. The sponge was removed and transferred repeatedly to 0.1 M hydrochloric acid until no colored byproducts were observed. The sponge was then immersed several times in ethanol to remove water and then left to dry in air at room temperature.

The coating with polypyrrole in the dispersion mode was carried out in a similar manner, and only water was replaced with a 2 wt % aqueous solution of PVP (molecular weight 360,000; Fluka, Switzerland) or 5 wt % colloidal nanosilica Ludox AS-40 (Du Pont product distributed by Aldrich).

**4.2. Characterization.** Morphology of sponges was observed with a scanning ultrahigh-resolution electron microscope MAIA3 Tescan (Brno, Czech Republic) and that of colloidal particles was observed with a transmission electron microscope Tecnai G2 Spirit (FEI).

FTIR spectra were recorded with a Thermo Nicolet 6700 FTIR spectrometer equipped with a GladiATR (PIKE Technologies, USA) using an attenuated total reflection (ATR) monolithic diamond in the wavenumber range 2000–400 cm<sup>-1</sup>, 64 scans at 4 cm<sup>-1</sup> resolution. The spectra were corrected for carbon dioxide and humidity in the optical path.

The specific surface area and pore volume were determined with a NOVA3200 (Quantachrome Instruments). Sponges were degassed for 24 h at 100 °C. The adsorption and desorption isotherms were recorded with nitrogen (Linde, 99.999%). Brunauer–Emmett–Teller analysis has been used to determine the total surface area, and the Barrett–Joyner–Halenda model was used for pore volume. Each sample was characterized four times, and the results were averaged. The experimental error was 5%.

For the electrical characterization, cylinders 10 mm in diameter were cut with a circular cork-cutter knife from the sponge and trimmed to ca 8 mm length. The conductivity was determined by the four-point van der Pauw method. A sample was placed in a lab-made cylindrical glass cell with an inner diameter of 10 mm between an insulating support and a glass piston carrying four platinum/rhodium electrodes on the perimeter of its base. The pressure applied to the sponge was controlled with an L6E3 load cell (Zemic Europe BV, The Netherlands). The thickness of the sponge during the compression was determined with a displacement meter Digimatic Indicator 543-122FB (Mitutoyo Corp., Japan).

For the determination of resistance in another apparatus, a cylinder having 19.5 mm diameter and 60 mm length was cut from the sponge. The sponge cylinder was placed inside the transparent tube of the same inner diameter directing the compression and expansion of the sponge. Two phosphor bronze circular electrodes were placed on the bottom and on the top of the sponge in the cylinder. A four-wire setup allowing us to measure low resistances have been used for continuous AC measurement (voltage 1 V, frequency 1 kHz) of parallel resistance  $R$  of the sample with an LCR-6002 meter (GW-Instek, USA). The pressure sensor was placed under the Sauter TVL testing stand equipped by force gauge Sauter

FH50 creating a maximum force of 50 N with a resolution of 0.01 N. The extent of compression and expansion was measured with an inbuilt deflection gauge with a resolution of 10 μm. The pressure  $P$  acting on the sensor was calculated using the formula  $P = F/A$ , where  $F$  is the force measured by the force gauge and  $A$  is an in-plane cross-sectional area of the sponge.

**4.3. Antibacterial Tests.** The antibacterial activity was determined using two common model bacterial species, the *Staphylococcus aureus* (CCM 4516) and *Escherichia coli* (CCM 4517) (both purchased from the Czech Collection of Microorganisms; Czech Republic). First, the bacterial suspension containing *S. aureus* or *E. coli* in nutrient broth (NB) and plate count-agar (PCA) (Sigma-Aldrich) was prepared according to 1/500 NB 8.4 g per 400 mL and PCA 9.4 g per 400 mL. The pieces of sponge of an external area of ~350 mm<sup>2</sup> were immersed in the bacterial suspension and gently shaken for 24 h at 37 °C. Thereafter, the bacterial suspensions from each sample were pipetted into plastic tubes subject to decimal dilutions. The individual solutions with the bacterial suspensions were homogenized, and 1 mL was pipetted onto agar. Samples were incubated for 24 h at 37 °C, and subsequently, the number of colonies (CFU mL<sup>-1</sup>) was evaluated using the computer-aided cell colony counting.

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### Notes

The authors declare no competing financial interest.

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