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Surface and antimicrobial properties of semi-fluorinated quaternary ammonium thiol surfactants potentially usable for Self-Assembled Monolayers

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ABSTRACT

The surfactant and antimicrobial activities of thiol and disulfide derivatives containing a quaternary ammonium group bearing variable perfluorinated carbon chains via an amide connector between the sulfur and nitrogen atoms were evaluated with the future aim to be grafted on metal surfaces for obtaining contact-active and non-adhesive auto-biocidal surfaces. Their biostatic and bactericidal activities against four microbial strains (*Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Aspergillus niger* and *Candida albicans*) were measured. The presence of the thiol, disulfide and amide functions in these surfactants were discussed in relation with antimicrobial activity along with the influence of the length of fluorinated chains in order to determine which molecular parameters are 'critical' for biological activity.

1. Introduction

The battle against nosocomial infections such as, among others, surgical infections, remains one of the major actual challenges of the hospital [1,2]. If cautions are numerous to avoid any pollution of inert surfaces (catheters, implants, medical equipments, floors...), the phenomena of resistance developed by the most part of pathogenic organisms require, on one hand, the elaboration of new biocide agents and, on the other hand, completion of long-term bactericidal treatments of surface or, in an ideal case, a permanent biocide and/or anti-adhesive effect of the surfaces without realising the surface active agents [1]. These surface treatments must avoid, on one hand, the contamination of the environment and the host, as well as short duration of antimicrobial action due to rapid releasing of active agents from the surface and, on the other hand, the formation of a biofilm of alive microorganisms [3,4]. Thus, the presence of covalently bonded biocides to the surfaces can avoid the biofilm formation. However, the killed microorganisms generally remain on the surface and, without washing, the formation of a new biofilm leads to the rapid decrease and then the disappearance of the biocide effect [5].

Concerning metallic materials, surfaces that could avoid the primary biofilm formation by killing harmful microorganisms on contact without releasing antimicrobial agents are in a growing field of research from a long time [6–9]. Among the different methods experimented, the concept of Self-Assembled Monolayers (SAMs) [10] of thiol and disulfide molecules covalently bound to metals and bearing potentially biocide moieties has been considered and promising results obtained [8,9]. However, the problem of removing the killed microorganisms from the surface, and consequently that of the persistence of the antimicrobial activity overtime, generally remain [11]. This loss of activity after a relatively short time could be considered as a non-negligible drawback of this SAMs strategy using short biocide structures. Some authors propose to elaborate anti-biofilm surfaces that combine the antimicrobial effect with a sort of continuous flow which would remove killed bacteria from the surface [9]. For our part, we propose to avoid the formation of the film of killed microorganisms by replacing the hydrocarbon chain on our biocide

Abbreviations: CMC, Critical Micellar Concentrations; MIC, Minimal Inhibitory Concentration; MLC, Minimal Lethal Concentration; SAMs, Self-Assembled Monolayers.

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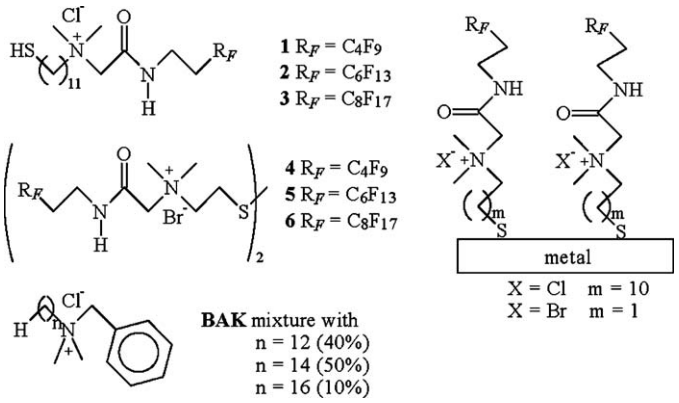

Scheme 1. Mono- and bis-ammonium salts studied, reference BAK 50, and corresponding SAMs on metal surfaces.

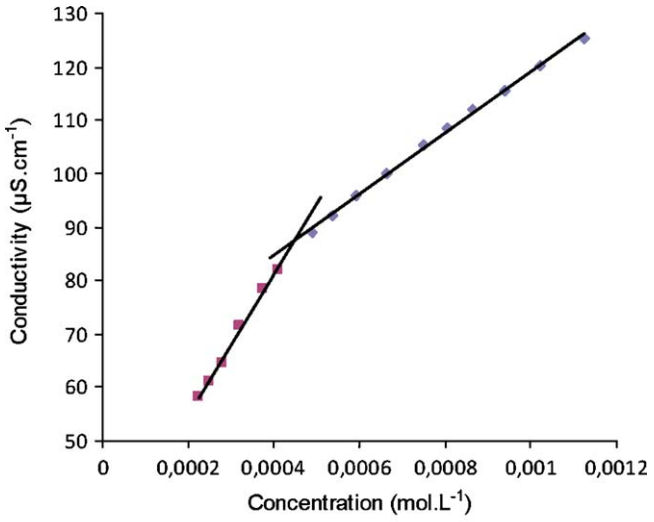

Fig. 1. Variations of the specific conductivity *K* with the surfactant concentration. *C* for 5 taken as example at 25 °C. CMC values of 4.5×10^{-4} mol l^{-1} were obtained from the intercept of both the *K* linear plots.

structures with perfluorinated alkyl chain. Indeed on one hand, our previous works showed that the presence of a perfluorinated chain can enhance the bactericidal activity of free quaternary ammonium salts [12–14] and, on the other hand, it is well known for a long time that perfluorinated coatings present anti-adhesive properties [15–17]. Thus, we can expect that these essential properties of perfluorinated chains could decrease the rate of formation of the "biopassivation" layer on the antimicrobial SAMs, and consequently lead to an increase of their biocidal efficiency overtime.

As the first step of this strategy, we demonstrated recently that, as simple semi-fluorinated alkanethiols R_F–R_H–SH or disulfides R_F–R_H–S–S–R_H–RF, perfluorinated thiols and disulfides bearing internal quaternary ammonium and amido groups in their structure, such as compounds **2**, **3**, **5** and **6**, can absorb on clean gold surface and form organized SAMs as shown in Scheme 1 [18]. So, the aim of the present work is to perform the second step of the strategy by testing the bactericidal efficiencies of two series of thiols **1**–**3** and disulfides **4**–**6** in solution, comparatively to the commercial available preservative BAK50 (50%, w/w aqueous solution of BAK) taken as reference, in view to obtain disinfecting antimicrobial surfaces efficient overtime after grafting on various metal surfaces. Moreover, the surfactant properties of these compounds will be evaluated and eventual relationships between these surface properties and the antimicrobial ones will be researched, since it is well known for a long time that, on one hand, the anti-adhesive effect of perfluorinated chains, as their surfactant effect, is closely related to the chain length and, on the other hand, the first effect used to explain the preservative effect of quaternary ammonium salts is due to their surfactant properties [19–24].

2. Results and discussion

2.1. Synthesis

Surfactants **1**–**6** were prepared in our laboratory according to the procedure reported previously [18]. The final yields of the synthesis obtained from the starting materials are reported in Table 1. The spectral and physico-chemical characteristics of new compounds **1** and **4** are summarized in experimental part.

2.2. Surface active properties

The Critical Micellar Concentrations (CMCs) were obtained by electrical conductivity measurements at 25 °C using the classical method described by Zana [25]. After determination of the CMC value of each surfactant as the intercept of both linear plots of conductivity *K* versus concentration of surfactant solutions (see for instance Fig. 1 for compound **5**), new solutions were prepared at the CMC for each surfactant and their surface tensions (γ_s at the CMC) were determined at 25 °C by the Wilhelmy plate method [26–28]. The substrates being insoluble in pure water, a 10% methanolic aqueous solution was used. It was demonstrated that the CMCs varies only slightly with 10% of methanol in water [29]. Fig. 2 that represents the variation of the surface tension at the CMC as a function of time for all the compounds show that rather long times are necessary to get the equilibrium state, particularly for disulfides **4** and **5**, and justifies the use of the conductimetry method rather than the tensiometry one to obtain relatively easily the CMC values.

The calculation of the micellization free energy, ΔG^0_M, associated with the transfer of the surfactant from the aqueous phase to the micellar pseudophase, is carried out from the general equations suggested by Zana [30]. For the thiols **1**–**3**, the standard free energy of the micellization can be obtained from the equation, $\Delta G^0_M = RT(2-\alpha)\ln(CMC)$, while, for the disulfures **4**–**6** that are "Gemini" surfactants the following simplified equation is used,

Table 1
Overall yields, Critical Micellar Concentration, surface tension at the CMC recorded at 25 °C, and micellization parameters α_{app} and ΔG^0_M for the surfactants **1**–**6**.

Surfactants	Overall yield (%)	10^{-4} CMC (mol l^{-1})	γ_{CMC} (mN m^{-1})	α_{app}	ΔG^0_M (kJ mol^{-1})
1	59[a]	6.3	26.0	0.67	−24.30
2	69[a]	2.9	22.3	0.71	−26.04
3	64[a]	1.0	20.2	0.62	−31.54
4	54[b]	11.0	21.9	0.45	−17.73
5	61[b]	4.5	18.7	0.43	−20.44
6	58[b]	1.2	16.8	0.42	−24.17

[a] From the starting material 11-bromo-undec-1-ene [18].
[b] From the starting material cystamine dihydrochloride [18].

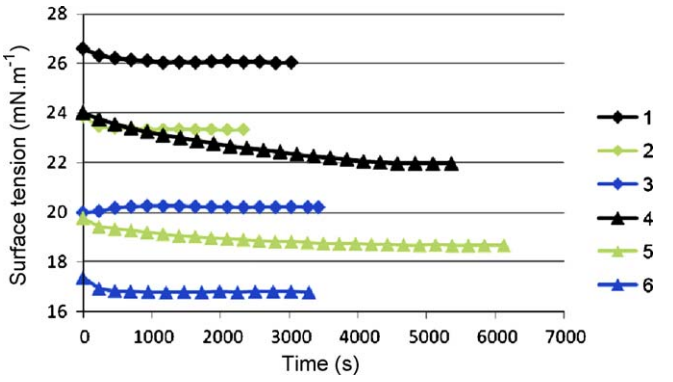

Fig. 2. Variation of the surface tension at the CMC as a function of time for **1**–**6**.

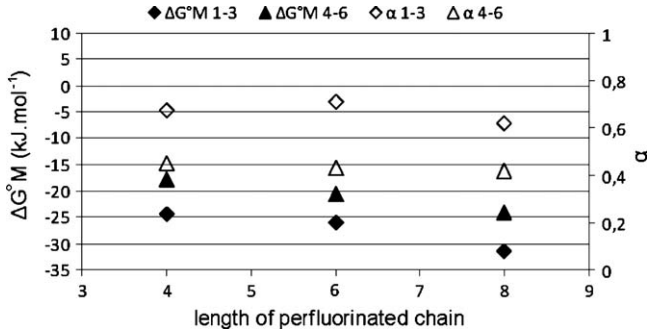

Fig. 4. Evolution of ΔG^0_M and α as a function of perfluorinated chain length for compounds **1**–**3** and **4**–**6**.

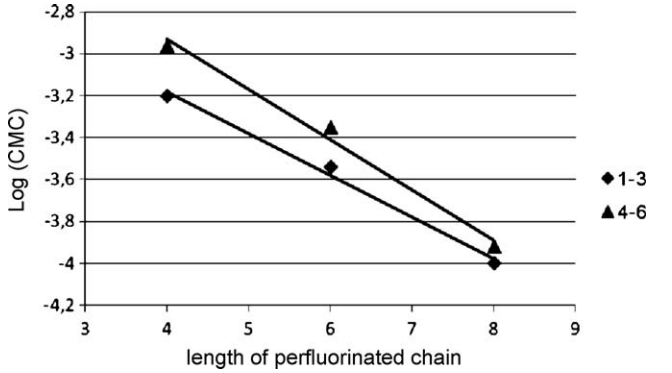

Fig. 3. Evolution of Log(CMC) as a function of perfluorinated chain length for **1**–**3** and **4**–**6**.

$\Delta G^0_M = RT(3/2 - \alpha)\ln(CMC)$, in which α is the degree of micelle ionization. Here, α is taken as the ratio of the values of $d\kappa/dC$ (κ denotes the conductivity) above and below the CMC obtained from the electrical conductivity measurements. The thermodynamic parameters of micellization α and ΔG^0_M for the semi-fluorinated surfactants **1**–**6** are collected in Table 1.

As expected in comparison with alkylated ammoniums or bis-ammoniums, these semi-fluorinated surfactants exhibit lower CMCs and γ_s, the surface tensions being lower for the Gemini surfactants **4**–**6** than for the mono-ammonium salts **1**–**3**. That could be explained by the presence of twice more fluorine in the case of Gemini. These values decrease when the length of perfluorinated chain increases as shown in Fig. 3. As expected, the slope of the curve for the bis-ammonium salts bearing two perfluorinated chains is greater than that of mono-ammonium salts.

In the same way, the decrease of the ΔG^0_M values with the perfluorinated chain length is related to the aggregation (micellization) which is favored, accompanied by a decrease of the CMC values (Fig. 3). $\Delta G^0_t(CF_2)$ can be defined as the slope of the line $\Delta G^0 = f$ (length of the fluorinated chain) and represents the free energy increment for the transfer of a fluoromethylene group from the aqueous phase to the micellar pseudophase. $\Delta G^0_t(CF_2)$ obtained for both series are very close and negative (−1.61 and −1.81 for the mono-ammonium salts **1**–**3** and Gemini surfactants **4**–**6**, respectively); it indicates that the energy gain is favorable when a CF_2 is added so that the micellization is privileged, which is consistent with the literature [31–33] (Fig. 4).

2.3. Antimicrobial activity

Antibacterial and antifungal evaluations of salts of the series **1**–**3** and **4**–**6** were run using MIC (Minimal Inhibitory Concentration) and MLC (Minimal Lethal Concentration) measurements versus four microorganisms: *Staphylococcus aureus* (ATCC 6538), *Pseudomonas aeruginosa* (ATCC 9027), *Aspergillus niger* (ATCC 6275) and *Candida albicans* (ATCC 10231). The strains used are ubiquitous, opportunistic and commonly encountered in biodeterioration material problems or fouling of the industrial processes, in addition these types of strains could be encountered in nosocomial infections. Table 2 summarizes the MIC values taken as an evaluation of the microbiostatic activity of all the molecules tested along with their MLC values taken as an evaluation of the microbicidal activity of these antimicrobial agents. Three independent experiments were undertaken for each compound.

In contrast with BAK 50 taken as standard, the semi-fluorinated mono-ammonium salts **1**–**3** present a microbiostatic activity only for *S. aureus*, while the Gemini surfactants **4**–**6** are active against the four microorganisms studied with the exception of compound **6** which shows only an acceptable activity towards *S. aureus*. These activities are always lower than for the alkylated ammoniums mixture BAK 50. In both series **1**–**3** and **4**–**6**, the highest activity is exhibited by the surfactants with $R_F = C_6F_{13}$. Thus, no direct dependence is observed between the surface properties CMCs or γ_s and the microbiostatic activity. However, the low CMC values for

Table 2
MIC ($\mu mol\,l^{-1}$) and MLC ($\mu mol\,l^{-1}$) values of surfactants **1**–**6**.

	Staphylococcus aureus		*Pseudomonas aeruginosa*		*Candida albicans*		*Aspergillus niger*	
	MIC	MLC	MIC	MLC	MIC	MLC	MIC	MLC
BAK 50	3.2		93		8.36		4.74	
1	56.1	>789.3[a]	>789.3[a]	>789.3[a]	>789.3[a]	>789.3[a]	>789.3[a]	>789.3[a]
2	13.6	37.6	>419.4[a]	>419.4[a]	>419.4[a]	>419.4[a]	>419.4[a]	>419.4[a]
3	83.9	>835.2[a]	>835.2[a]	>835.2[a]	>835.2[a]	>835.2[a]	>835.2[a]	>835.2[a]
4	27.8	30.2	145.5	145.5	54.3	146.8	40.7	>56.5[a]
5	17.4	23.8	98.4	98.4	62.1	145.2	20.6	>97.7[a]
6	102.1	>423[a]	>423[a]	>423[a]	>423[a]	>423[a]	>423[a]	>423[a]

[a] No activity detected, the values given are the maximal detectable concentrations of antimicrobial agents.

these semi-fluorinated ammonium salts can explain the loss of activity of compounds **1**–**3** and **6** towards *P. aeruginosa*, *C. albicans* and *A. niger*. Indeed, it has been shown that the solution behaviour of quaternary ammonium surfactants affects their antimicrobial activity [34–38]. Now the CMC represents the highest concentration of "free" surfactant molecules in solution and a micelle represents a form less active of the surfactant than the "free" surfactant molecule. Thus when the CMCs are too low, it is impossible to get the MIC values for the "free" surfactant. Concerning the series **4**–**6** for which the antimicrobial activity is higher and the CMC also slightly higher than for series **1**–**3**, MIC values towards *P. aeruginosa*, *C. albicans* and *A. niger* can be got with the exception of **6** that presents the highest MIC value for *S. aureus* and the lowest CMC in the series.

These observations can be applied to the microbicidal activities represented by the MLC values. Only, Gemini surfactants **4** and **5** show MLC values for *S. aureus*, *P. aeruginosa*, and *C. albicans*, the compound **5** bearing the C_6F_{13} chain showing the highest microbicidal activity. The impossibility of measuring MLC values for the other compounds is not surprising. Indeed in agreement with the literature, the MLC values are always greater than the MIC values since the frontier between microbicidal and microbiostatic activities is often a question of concentrations used; some compounds can be microbiostatic at low concentration and microbicidal at higher concentrations [19].

3. Conclusion

The two series, **1**–**3** and **4**–**6**, of simple and double tailed cationic surfactants studied in this work exhibit low CMC and surface tension values. The presence of the thiol function at the end of the long hydrocarbon chain in compounds **1**–**3**, does not affect greatly the surface properties. As expected, the surface activity increases with the length of the perfluorinated chain. These compounds show interesting antimicrobial activities against *S. aureus*, but only moderate (for the Gemini surfactants) to poor (for the mono-ammonium salts) activities against the other strains studied. For both series, the best efficiency is observed for the surfactants bearing the intermediate C_6F_{13} perfluorinated chain. Thus no directed relationships can be observed between the antimicrobial activities and the surface parameters. The future elaboration of contact-active autobiocidal and anti-adhesive metal surfaces via SAMs formation on metal surface using these semi-fluorinated thiols and disulfures can be envisaged. Nevertheless, we can expect a biocidal surface activity only for *S. aureus* that constitutes an important limitation of our strategy.

4. Experimental

4.1. Chemical synthesis

Surfactants **1**–**6** were prepared in our laboratory according to the procedure reported previously [18]. ^{1}H and ^{13}C Nuclear Magnetic Resonance (NMR) spectra were recorded on a Brüker AC 200 spectrometer. Signal attributions were confirmed by HMQC (2D NMR) experiments. The mass spectra were recorded on a Finnigan Mat LCQ Classic Electrospray source API1. Elemental analyses were performed using a Flash EA1112 automatic elemental analyzer from Thermo Scientific equipped with Eager 300 software. The spectral and physico-chemical characteristics of compounds **2**, **3**, **5** and **6** were previously described.

Compound **1**: ^{1}H NMR (200 MHz, MeOD): δ (ppm) 1.24 (m, 14H [$-CH_2-$]$_7$), 1.61 (m, 2H [SCH_2-CH_2]), 1.80 (m, 2H [$N^+-CH_2-CH_2$]), 2.44 (m, 4H [$S-CH_2$ and CH_2-CF_2]), 3.21 (s, 6H, [$N^+(CH_3)_3$]), 3.50 (m, 4H [$NH-CH_2$ and N^+-CH_2]), 4.06 (s, 2H [$CH_2-C(O)$]).

^{13}C NMR (MeOD): δ (ppm) 23.8 (N–C–*C*), 25.2 (S–*C*), 30.0 (S–C–C–C_7), 31.2 (*C*–CF_2), 33.1 (NH–*C*), 35.5 (S–C–*C*), 53.2 (N–CH_3), 62.7 (*C*–C(O)), 65.0 (N^+–CH_2), 165.3 (*C*=O).

19F NMR (MeOD): δ (ppm) −83 (CF_3), −116.3 (CF_2), −125.0 (CF_2), −127.1 (CF_2).

MS, $[M-Cl]^+$, *m*/*z* (%): 535 (100). Anal. Calcd for $C_{21}H_{36}F_9N_2OSCl$: C 44.17, H 6.35, N 4.91, S 5.61; found: C 44.07, H 6.51, N 4.97, S 5.63.

Compound **4**: ^{1}H NMR (200 MHz, MeOD): δ (ppm) 2.51 (tt, 4H, J^3_{H-H} = 7.0 Hz, J^3_{H-F} = 19.6 Hz [CF_2-CH_2]), 3.41 (m, 16H [$S-CH_2-$]$_7$ and [$N(CH_3)_2$]), 3.57 (t, 4H, J^3_{H-H} = 7.0 Hz [$NH-CH_2$]), 4.08 (m, 4H [N^+-CH_2]), 4.27 (s, 4H [$CH_2-C(O)$]).

^{13}C NMR (MeOD): δ (ppm) 30.8 (CF_2–*C*), 32.2 (S–*C*), 33.4 (NH–*C*), 52.7 (N–CH_3), 63.1 (N^+–*C*–CO), 65.8 (S–C–*C*), 166.0 (*C*=O).

19F NMR (MeOD): δ (ppm) −83 (CF_3), −116.3 (CF_2), −125.0 (CF_2), −127.1 (CF_2).

MS, $[M-2Br]^{2+}/2$ (%): 408.1 (100). Anal. Calcd for $C_{24}H_{34}F_{18}N_4O_2S_2Br_2$: C 29.52, H 3.51, N 5.74, S 6.57; found: C 29.81, H 3.53, N 5.66, S 6.63.

4.2. Surfactant properties evaluation

The CMC were determined from conductimetry measurements at 25.0 ± 0.1 °C using a Consort C831 conductimeter. The measurement of conductivity was carried out with an absolute accuracy up to ±3%. The solutions were prepared by weight using an electronic balance with an accuracy of $\pm 1 \times 10^{-4}$ g. The repeatability of the conductivity measurements, estimated from two successive runs, was about ±0.5%. The surface tensions at the CMC were determined at 25 °C using a tensiometer Krüss K100. The substrates being insoluble in pure water, a 10% methanolic aqueous solution was used.

4.3. Antimicrobial characteristics

4.3.1. MIC determination

MIC is defined as the lowest concentration of an antimicrobial that will inhibit the visible growth of a microorganism after overnight incubation. The MICs were taken as the minimal concentration showing no growth (absence of turbidity) after 24 h of incubation at 30 °C for bacteria and after 72 h of incubation at 25 °C for fungi. MICs were determined using a serial dilution method. The automat Biomek® 1000 (Beckman®) apparatus used for our experiments performed automatically dilutions of the tested antimicrobial agents solutions in 96 well micro-titration plates.

4.3.2. MLC determination

The MLC is defined as the lowest concentration of antimicrobial that will prevent the growth of a microorganism after subculture onto antibiotic-free media. The MLC levels were determined by taking (after 5 days incubation for bacteria or 7 days for fungi) from MIC microtiter plates, 100 μl from the three sub-doses wells where no growth was observed, and subculture by streaking onto trypticase soy agar (TSA) or sabouraud dextrose. Agar (SDA) plates are incubated as described below and checked after incubation period to determine whether or not viable microorganisms remain.

4.3.3. Cultivation

MIC determinations were run with the third generation of bacteria or fungi. For bacteria, samples were taken during the exponential phase. Bacteria were grown overnight on a TSA slant and inoculated into 20 ml of M9GY at pH 7.0 ± 0.2 culture medium and incubated overnight at 30 °C. The optical density of the suspension was then measured at 660 nm. If necessary, additional M9GY medium was added to the suspension to adjust the optical

density at 660 nm to 0.05 corresponding approximately to between 5×10^6 and 5×10^7 cfu/ml.

Fungi were incubated on SDA slants for 5 days at 25 °C. MIC determinations were run with the third generation of fungi; culture samples were taken during the exponential phase of fungal growth. Each fungal strain was cultivated during 5–7 days on SDA at 25 ± 2 °C until sufficient spores were formed. Then fungal spores were harvested by adding 5 ml of M9GY at pH 5.0 ± 0.2 to the SDA slant, which were then gently scraped to suspend the microorganisms. The fungal solution is then filtered with sterile cheesecloth under aseptic conditions to eliminate the residual mycelium. The suspension was adjusted using a counting cell under microscope, to 5×10^6 to 5×10^7 spores per millilitre by adding M9GY if necessary.

Initial solution concentration of each antimicrobial agent was prepared in a mixture of methanol/sterile deionised water (ratio 1/1).

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