

Uniform volumetric encapsulation of antimicrobial agents into mesoporous silica for their slow release: the case of copper oxide

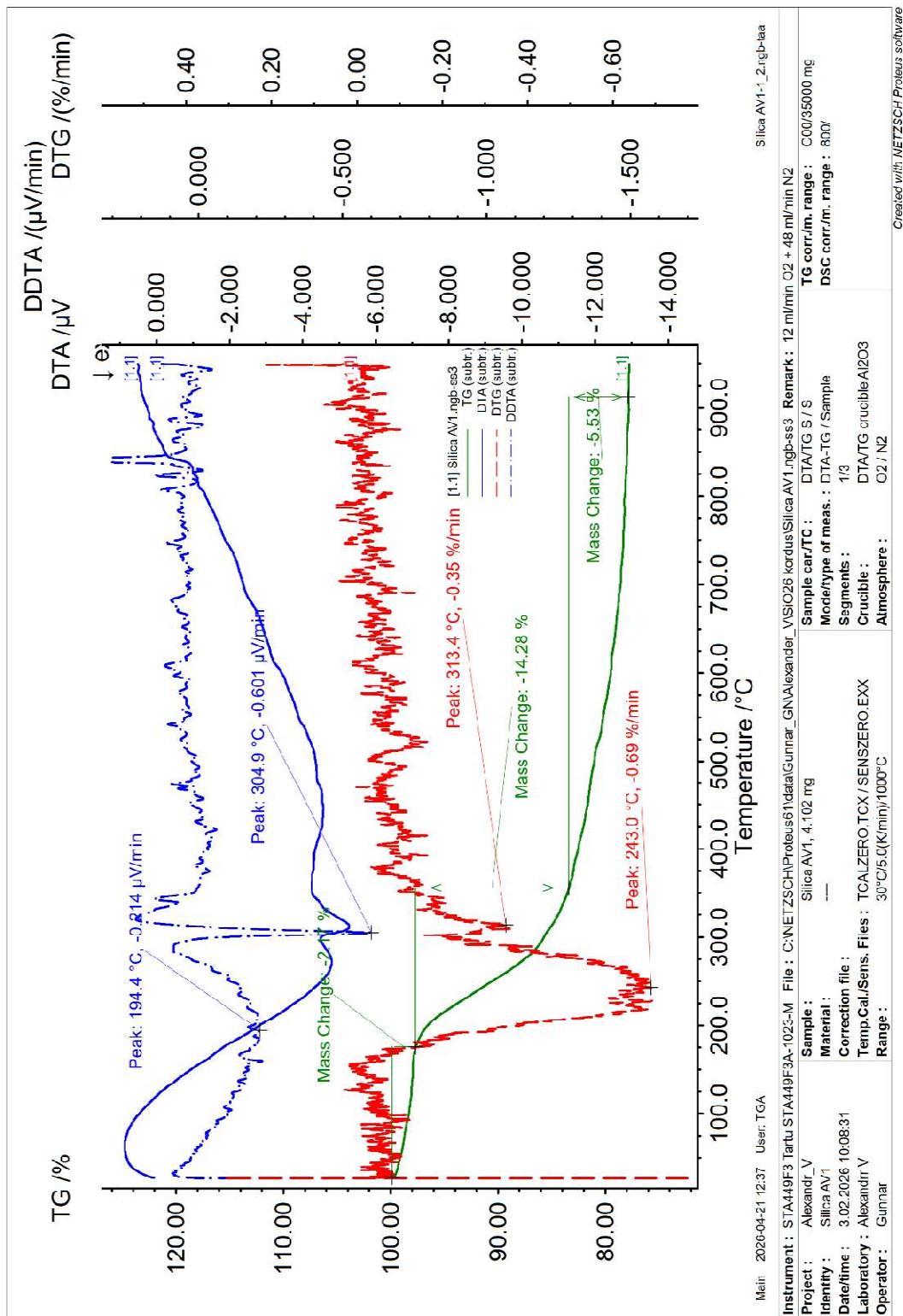
Aadil Shafi Bhat¹, Mariliis Sihtmäe², Kaja Kasemets², Mati Kook¹, Jekaterina Kozlova¹, Hugo Mändar¹, Alexandra Nefedova¹, Gunnar Nurk³, Angela Ivask⁴, Vambola Kisand¹, Alexander Vanetsev^{1*}

¹ Institute of Physics, University of Tartu, W. Ostwaldi 1, 50411 Tartu, Estonia

² National Institute of Chemical Physics and Biophysics, Akadeemia tee 23, 12618, Tallinn, Estonia

³ Institute of Chemistry, University of Tartu, Ravila tn 14A, 50411 Tartu, Estonia

⁴ Institute of Molecular and Cell Biology, University of Tartu, Riia 23, 51010 Tartu, Estonia



Mein: 2026-04-21 12:37 User: TGA
 Instrument: STA449F3 Tartu STA449F3A-1025-M File: C:\NETZSCH\Proteus61\data\Gunnar_GNA\Alexander_VISO26_kordus\Silica AV1.npb-ss3 Remark: 12 ml/min O2 + 48 ml/min N2
 Project: Alexander_V Sample: Silica AV1, 4,102 mg
 Identity: Silica AV1 Material: ---
 Date/time: 3.02.2026 10:08:31 Correction file:
 Laboratory: Alexander V Temp.Cal./Sens. Files: TCALZERO.TCX / SENSZERO.EXX
 Operator: Gunnar Range: 30°C/5.0(K/min)/1000°C
 Sample car/TG: DTA/TG S7 S TG corr./m. range: C00/35000 mg
 Mode/type of meas.: DTA-TG / Sample DSC corr./m. range: 800Y
 Segments: 1/3
 Crucible: DTA/TG crucibleAl2O3
 Atmosphere: O2 / N2
 Silica AV1_1_2.npb-ss3
 Created with: NETZSCH Proteus software

Figure S1. DT/TGA of as prepared SiO₂ sample.

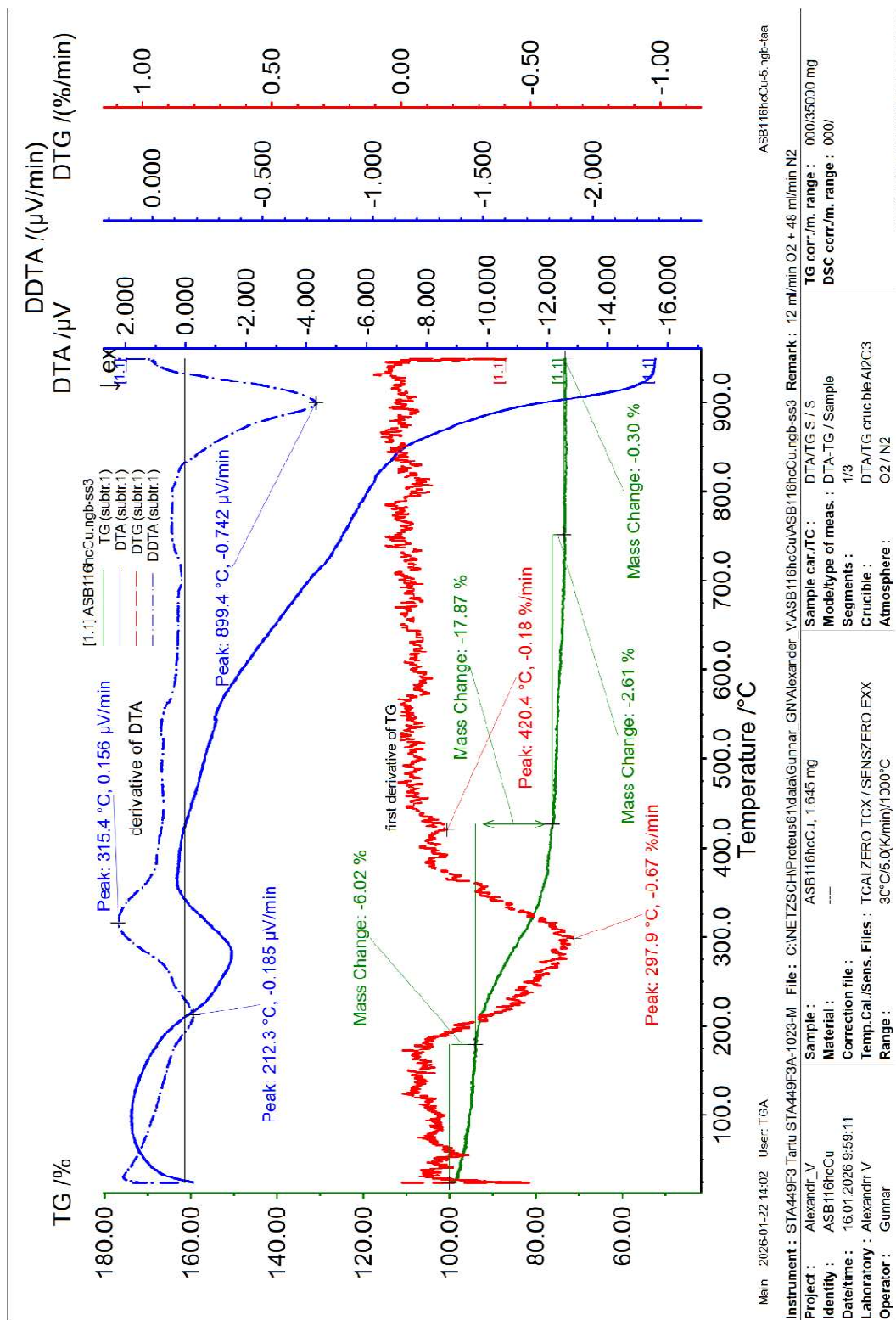


Figure S2. DT/TGA of $[\text{Cu}(\text{NH}_3)_4]^{2+}@\text{SiO}_2$ (5.5%) sample.

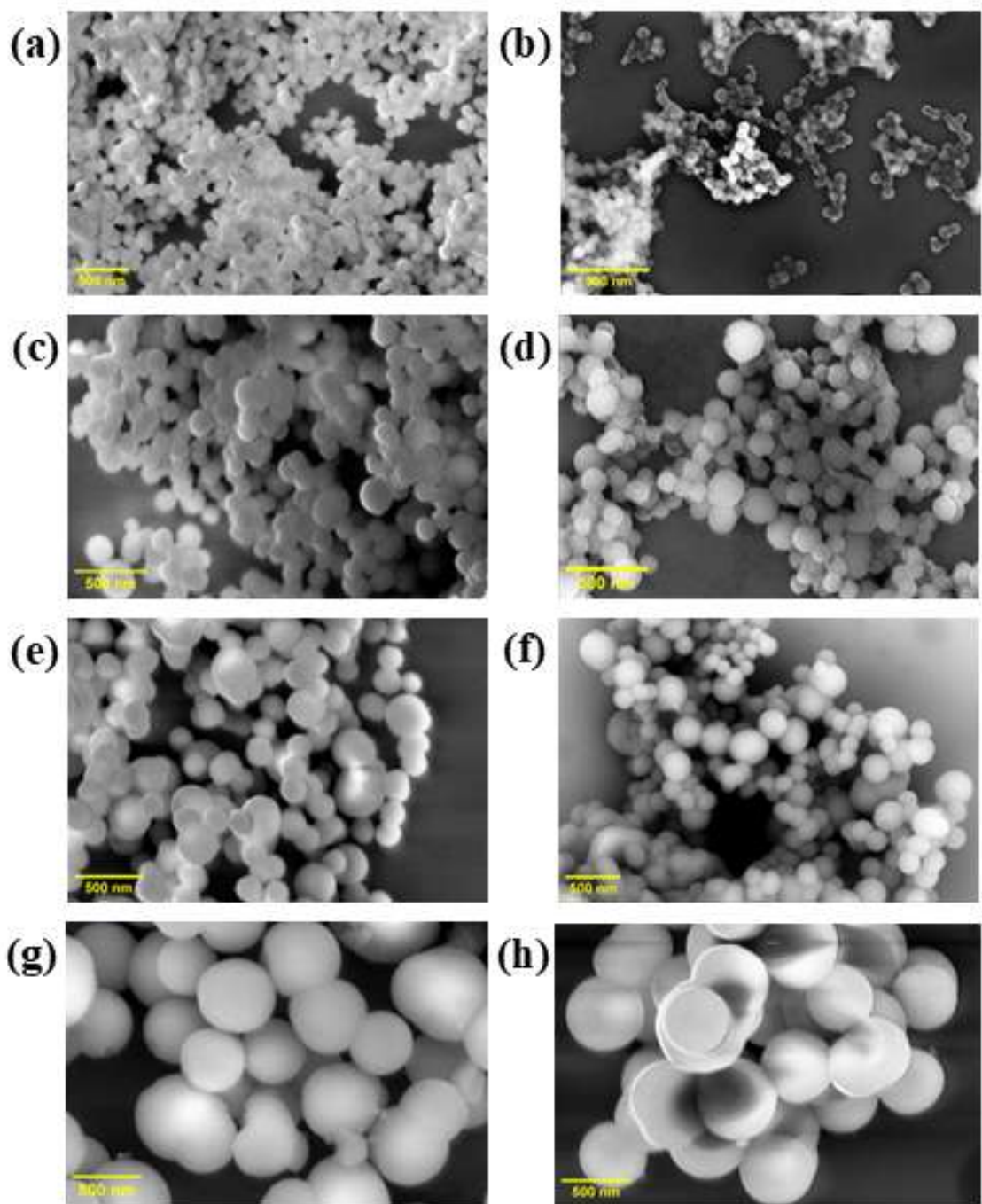


Figure S3. Scanning electron microscopy images of SiO₂ (a) and calcined SiO₂ (b); [Cu(NH₃)₄]²⁺@SiO₂ (1.5%) (c) and Cu_xO@SiO₂ (1.5%) (d); [Cu(NH₃)₄]²⁺@SiO₂ (3%) (e) and Cu_xO@SiO₂ (3%) (f); and [Cu(NH₃)₄]²⁺@SiO₂ (5.5%) (g) and Cu_xO@SiO₂ (5.5%) (h). Scale bars = 500 nm.

Table S1. Colony-forming ability of bacteria and yeast after 4-h and 24-h exposure to nanocomposites and respective positive controls (CTAB and CuSO₄·5H₂O) to bacteria and yeast in deionized water at 30°C. After exposure, cells (3 μL) were transferred onto toxicant-free agarized growth medium. All experiments were performed in at least three independent replicates; images shown are representative.

Compound or material	<i>E. coli</i> ATCC 25922: 4-h										<i>E. coli</i> ATCC 25922: 24-h									
	Concentrations: μg compound/mL (μg Cu/mL)										Concentrations: μg compound/mL (μg Cu/mL)									
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200
	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%)																				
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%) 24h leached																				
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200
	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)
Cu _x O@SiO ₂ (5.5%)																				
Cu _x O@SiO ₂ (5.5%) 24h leached																				
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200				
SiO ₂ , as prepared																				
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200				
SiO ₂ , calcined																				
	0	0.1	0.5	1.0	2.5	5	10	0	0.1	0.5	1.0	2.5	5	10						
CTAB																				
	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3		
	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)		
CuSO ₄ ·5H ₂ O																				

Compound or material	<i>P. aeruginosa</i> ATCC 27853: 4-h											<i>P. aeruginosa</i> ATCC 27853: 24-h										
	Concentrations: μg compound/mL (μg Cu/mL)											Concentrations: μg compound/mL (μg Cu/mL)										
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200		
	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)		
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%)																						
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%) 24h leached																						
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200		
	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)		
Cu _x O@SiO ₂ (5.5%)																						
Cu _x O@SiO ₂ (5.5%) 24h leached																						
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200						
SiO ₂ , as prepared																						
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200						
SiO ₂ , calcined																						
	0	0.1	0.5	1.0	2.5	5	10	0	0.1	0.5	1.0	2.5	5	10								
CTAB																						
	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3				
	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)				
CuSO ₄ ·5H ₂ O																						

Compound or material	<i>S. aureus</i> ATCC 6538: 4-h										<i>S. aureus</i> ATCC 6538: 24-h									
	Concentrations: μg compound/mL (μg Cu/mL)										Concentrations: μg compound/mL (μg Cu/mL)									
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200
	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)	0	(0.05)	(0.09)	(0.19)	(0.4)	(0.8)	(1.5)	(3)	(6)	(12)
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%)																				
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%) 24h leached																				
	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200	0	0.8	1.6	3.1	6.3	12.5	25	50	100	200
	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)	0	(0.05)	(0.1)	(0.2)	(0.4)	(0.8)	(1.6)	(3.2)	(6.4)	(12.8)
Cu _x O@SiO ₂ (5.5%)																				
Cu _x O@SiO ₂ (5.5%) 24h leached																				
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200				
SiO ₂ , as prepared																				
	0	3.1	6.3	12.5	25	50	100	200	0	3.1	6.3	12.5	25	50	100	200				
SiO ₂ , calcined																				
	0	0.1	0.5	1.0	2.5	5	10	0	0.1	0.5	1.0	2.5	5	10						
CTAB																				
	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3	0	0.20	0.4	1.0	2.0	3.9	9.8	19.6	39.3		
	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)	0	(0.05)	(0.1)	(0.25)	(0.5)	(1.0)	(2.5)	(5.0)	(10)		
CuSO ₄ ·5H ₂ O																				

Compound or material	<i>C. albicans</i> ATCC 10231: 4-h Concentrations: μg compound/mL (μg Cu/mL)								<i>C. albicans</i> ATCC 10231: 24-h Concentrations: μg compound/mL (μg Cu/mL)																							
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%)	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)
Cu _x O@SiO ₂ (5.5%)	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)
SiO ₂ , as prepared	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
SiO ₂ , calcined	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500
CTAB	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250
	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250	0	3.9	7.8	15.6	31.3	62.5	125	250
CuSO ₄ ·5H ₂ O	0	2.5	4.9	9.8	20	39	79	157	0	2.5	4.9	9.8	20	39	79	157	0	2.5	4.9	9.8	20	39	79	157	0	2.5	4.9	9.8	20	39	79	157
	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)

Compound or material	<i>C. auris</i> ATCC MYA 5001: 4-h Concentrations: μg compound/mL (μg Cu/mL)								<i>C. auris</i> ATCC MYA 5001: 24-h Concentrations: μg compound/mL (μg Cu/mL)								
[Cu(NH ₃) ₄] ²⁺ @SiO ₂ (5.5%)	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)	0	(0.5)	(0.9)	(1.8)	(3.6)	(7.3)	(15)	(29)	
Cu _x O@SiO ₂ (5.5%)	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)	0	(0.5)	(0.9)	(1.9)	(3.8)	(7.5)	(15)	(30)	
SiO ₂ , as prepared	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
SiO ₂ , calcined	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
	0	7.8	15.6	31.3	62.5	125	250	500	0	7.8	15.6	31.3	62.5	125	250	500	
CTAB	0	1.0	1.95	3.9	7.8	15.6	31.3	62.5	0	1.0	1.95	3.9	7.8	15.6	31.3	62.5	
	0	1.0	1.95	3.9	7.8	15.6	31.3	62.5	0	1.0	1.95	3.9	7.8	15.6	31.3	62.5	
CuSO ₄ ·5H ₂ O	0	2.5	4.9	9.8	20	39	79	157	0	2.5	4.9	9.8	20	39	79	157	
	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)	0	(0.6)	(1.3)	(2.5)	(5)	(10)	(20)	(40)	