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Application of reduced graphene oxide in the photolithography process of biodegradable composite to improve its electrical conductivity

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Abstract

The combination of single-walled carbon nanotubes (SWCNTs) and reduced graphene oxide (rGO) in the photoresist minimizes the amount of carbon particles. While developing neuroimplants, designed to restore damaged neural networks or modulate pain transmission, the key requirements are both biocompatibility and electrical conductivity.

Methods

Photolithography of the composite was performed with an ytterbium laser at a wavelength of 1035 nm with pulses of 100 ns duration, repetition rate of 30 kHz and power of ~550 mW (Fig. 1). The photoresist used was SWCNT 0.6 mg/mL, rGO 0.6 mg/mL and SWCNT (0.3 mg/mL)/rGO (0.3 mg/mL). The final biohybrid structure contains proteins, chitosan and eosin Y (Fig. 2).

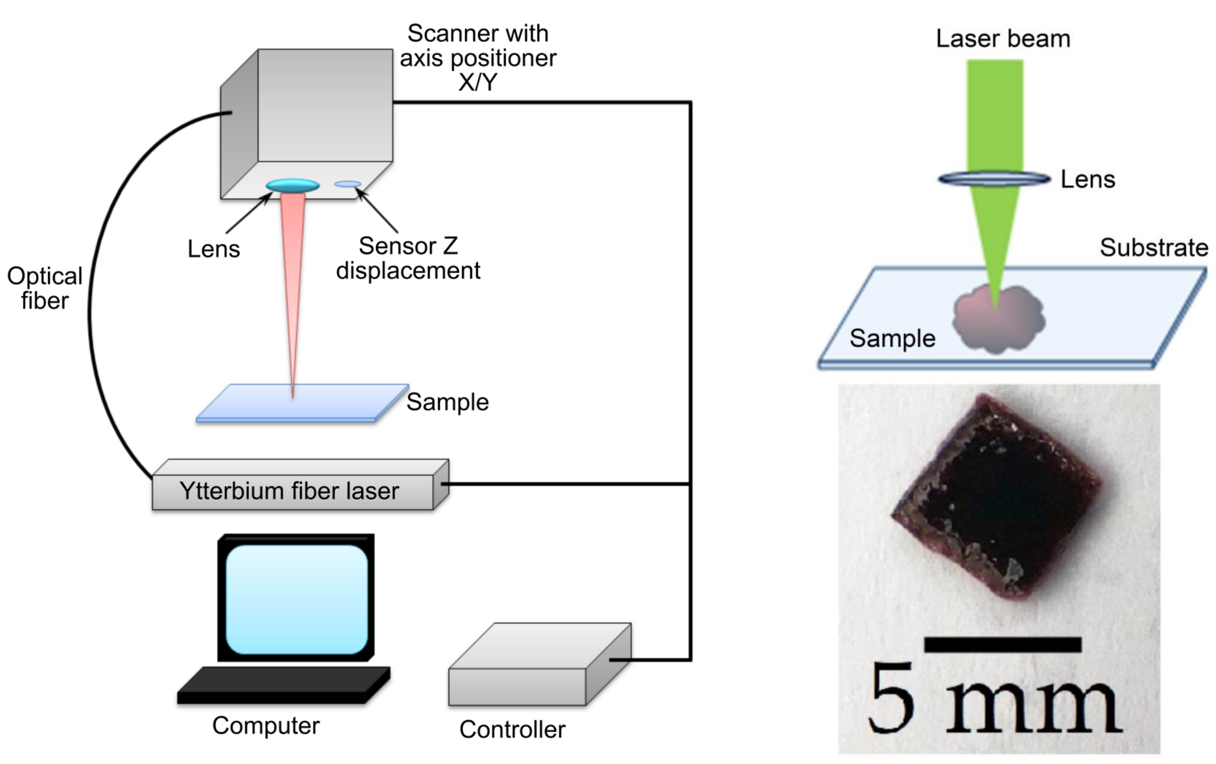
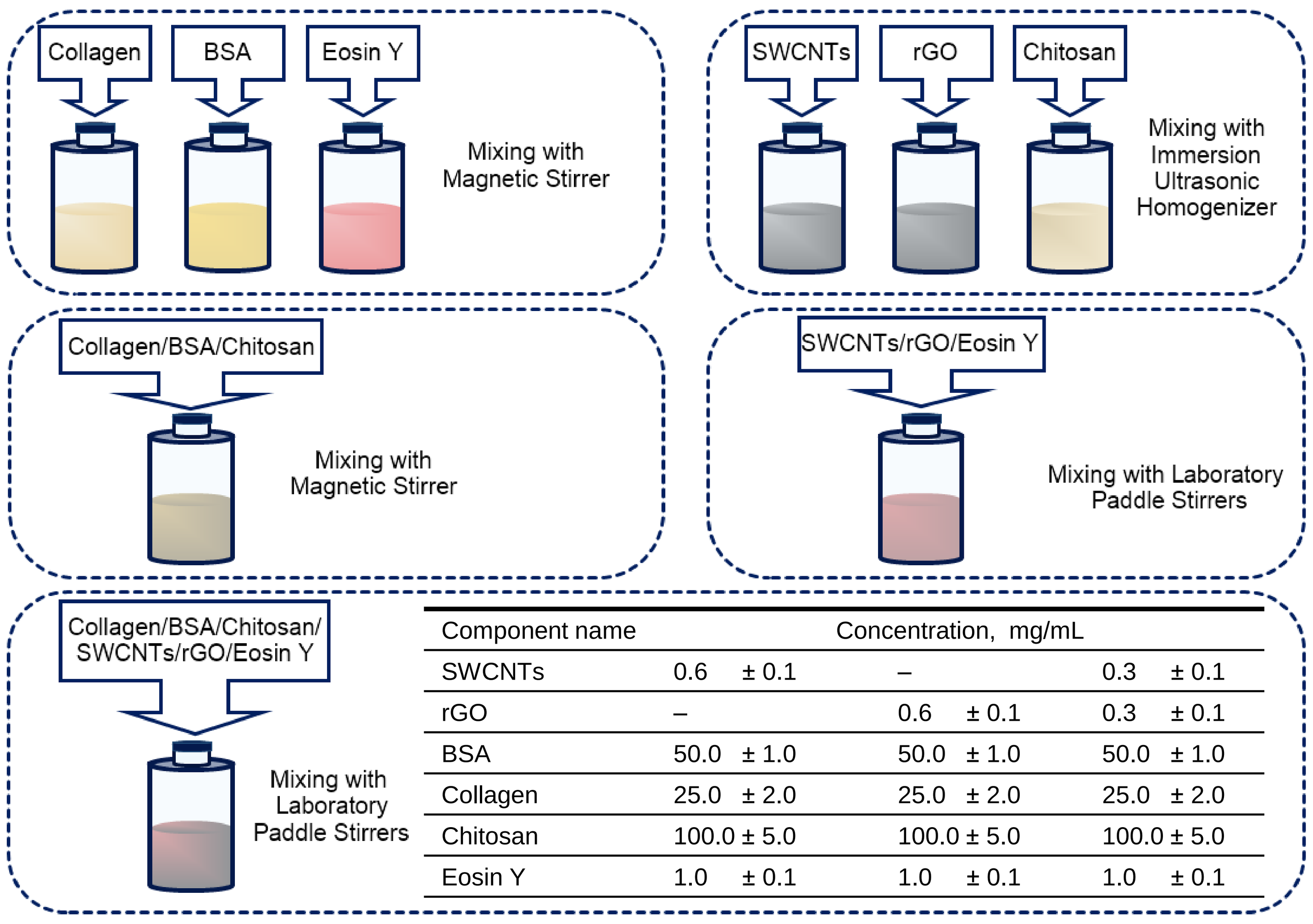


Figure 1. Laser system



Component name	Concentration, mg/mL		
SWCNTs	0.6 ± 0.1	–	0.3 ± 0.1
rGO	–	0.6 ± 0.1	0.3 ± 0.1
BSA	50.0 ± 1.0	50.0 ± 1.0	50.0 ± 1.0
Collagen	25.0 ± 2.0	25.0 ± 2.0	25.0 ± 2.0
Chitosan	100.0 ± 5.0	100.0 ± 5.0	100.0 ± 5.0
Eosin Y	1.0 ± 0.1	1.0 ± 0.1	1.0 ± 0.1

Figure 2. Composition of Photoresist

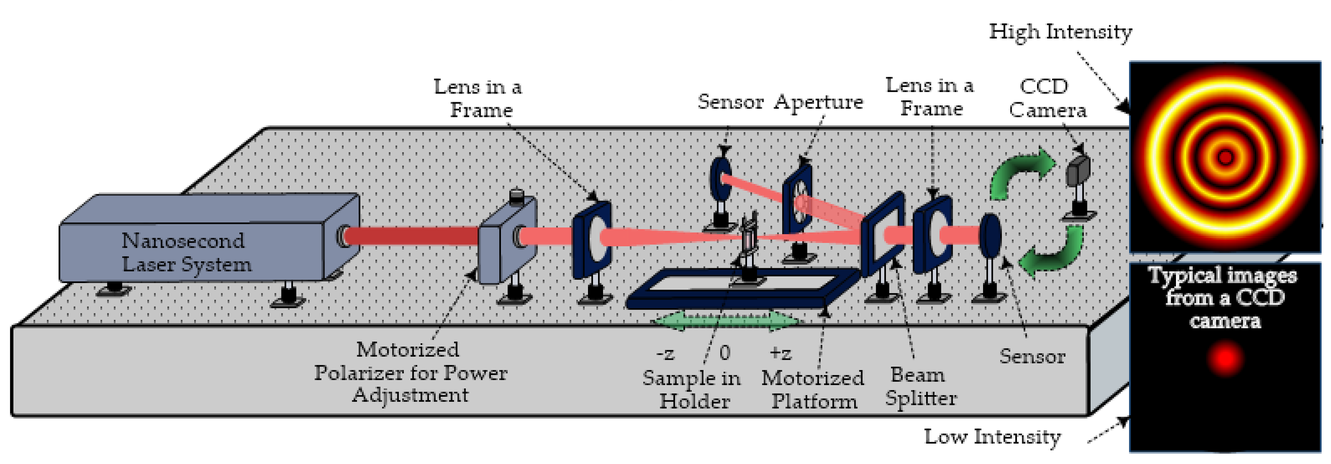


Figure 3. Scheme of the setup for conducting research using the Z-scan method

Results

The presence of appropriate optical properties for photopolymerization was assessed using the Z-scan method (Table 1). Four samples with different particle size distributions were prepared. The slide shows the spectra for each photoresist component. All of its components, with the exception of Eosin Y, have only insignificant linear absorption at a wavelength near 535 nm. It is its presence that allows photopolymerization using laser radiation with a wavelength of 1070 nm, which is confirmed by the absence of such an effect outside the focus of the lens.

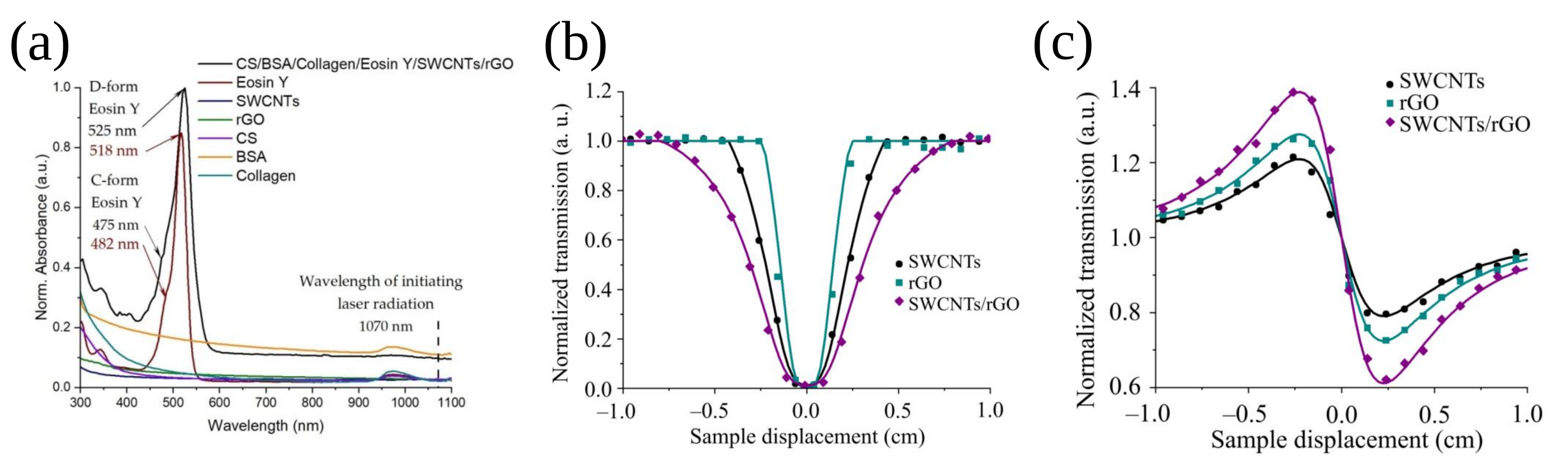


Figure 4. Results of measurements using the Z-scan with open aperture (a), close aperture (b) and spectra of photoresist components (c)

Table 1. Optical parameters of photoresist

Photoresist	SWCNTs concentration, mg/mL	rGO concentration, mg/mL	Linear Absorption Coefficient α, cm^{-1}	Nonlinear Absorption Cross Section, σ, GM	Threshold Exposure to Laser Radiation, F_x, J/cm^2	Linear Refractive Index, n_0, a.u.	Nonlinear Refractive Index, n_n, cm^2/TW
1	0.6	–	35	530	0.15	1.3619	15
2	–	0.6	25	430	0.25	1.3642	15
3	0.3	0.3	31	640	0.05	1.3628	25

The nonlinear absorption cross section was determined by the formula:

$$\sigma = \frac{\beta \hbar \omega}{N_A C \cdot 10^{-3}} \cdot 10^{50} (\mathrm{GM}). \quad (1)$$

where β is nonlinear absorption coefficient (cm/W); N_A is Avogadro's number (mol^{-1}); C is concentration moles divided by liter (mol/L); $\hbar$ is Dirac constant (J·s); ω is cyclic frequency (Hz). The nonlinear refractive index is determined according to the formula:

$$T = 1 - \frac{8\pi n_n I_0 \left(1 - \exp(-\alpha d)\right) x}{\lambda \alpha \left(x^2 + 9\right)\left(x^2 + 1\right)}. \quad (2)$$

where $x = z/z_0$ is normalized coordinate; α is linear absorption coefficient; d is optical thickness of the sample; I_0 is maximum intensity in focus; λ is wavelength; n_2 is nonlinear refractive index.

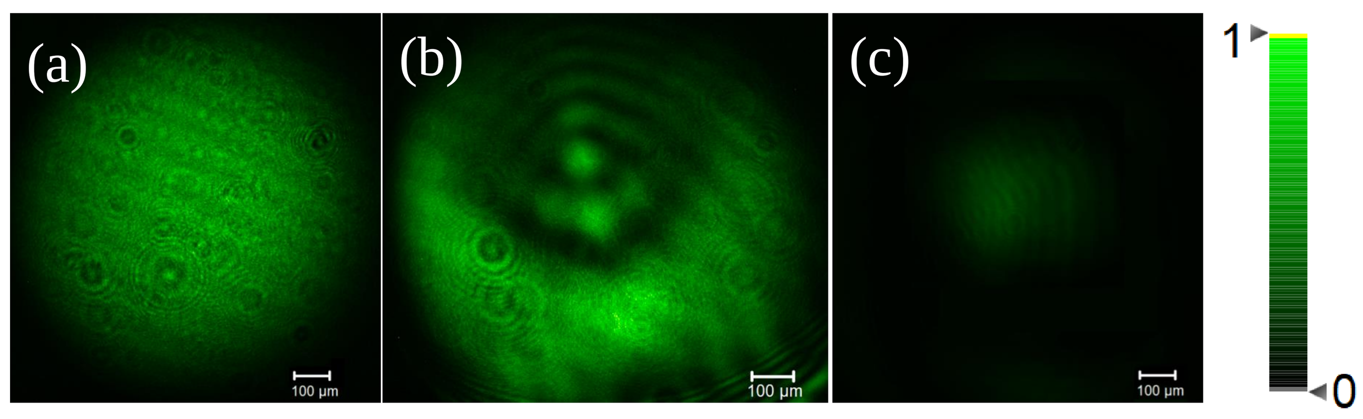


Figure 4. Beam profile when irradiating a sample with laser pulses: nanosecond duration far from focus (a), in focus (b) and with formed composite after 0.3 s (c)

The number of laser beam passes for stable polymerization is determined by the formula:

$$N = \frac{\vartheta \cdot t}{L}. \quad (3)$$

where ϑ is laser spot movement speed; t is the time from the start of laser irradiation until the disappearance of the diffraction ring pattern (Fig. 4); L is line length.

The formation of the structure was simulated by the molecular dynamics method with SEM monitoring (Fig. 5).

The specific electrical conductivity of 15 layers 5×5 mm was determined using the four-probe method (Table 2) and Fig. 6. Biodegradation was estimated by the mass of the swollen sample in an isotonic sodium chloride solution and dried, and enzymes: lipase - 25,000 PhEur; amylase - 18,000 PhEur and protease - 1000 PhEur (Fig. 7). In vitro biocompatibility studies were conducted with the Neuro 2A cell line with the MTT test for 72 hours (Fig. 8).

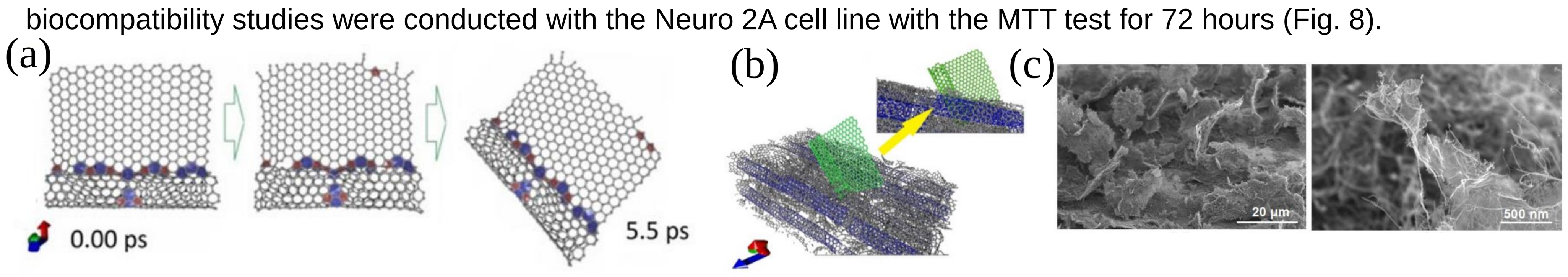


Figure 5. Response of a hybrid based on a graphene flake bonded to SWCNT to an external electric field over time (a), a fragment of the layer surface of hybrids based on graphene flakes bonded to SWCNT after laser exposure (b) and SEM images of layer structures based on rGO/SWCNT hybrids after laser exposure with an energy density of 0.5 J/cm^2(c)

Table 2. Electrical conductivity of samples

Biocomposites, №	SWCNTs concentration, mg/mL	rGO concentration, mg/mL	Specific Conductivity, mS×cm^{-1}
1	0.6	–	19
2	–	0.6	17
3	0.3	0.3	35

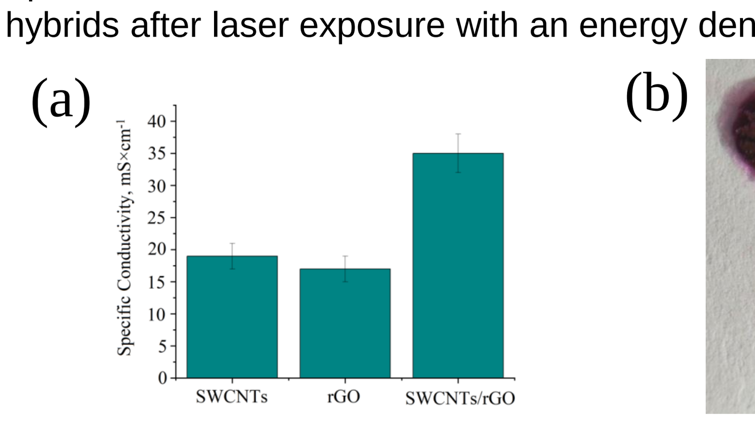


Figure 6. Specific conductivity of biocomposite (a) and appearance of biocomposites (b)

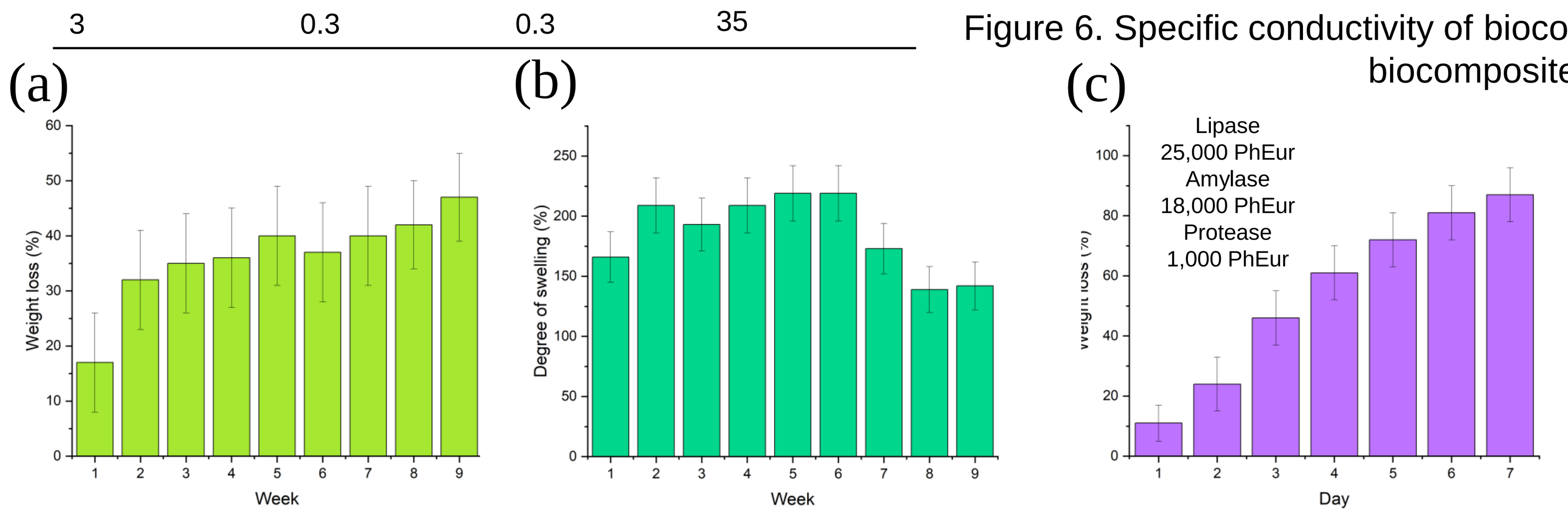


Figure 7. Mass loss in nine weeks (a), the degree of re-swelling relative to the initial (b) and mass loss in seven days with enzymatic degradation (c) (Collagen/BSA/Chitosan/SWCNTs/rGO/Eosin Y)

Estimation of mass loss and swelling degree

$$M_t = \frac{m_0 - m_t}{m_0} \times 100\%, \quad (4)$$

where M_t is the mass loss after t days of degradation, m_0 is the initial value of the mass of the dry sample before degradation, m_t is the mass after t days.

$$S_t = \frac{m_{wt} - m_t}{m_0} \times 100\%, \quad (5)$$

where S_t is the degree of re-swelling after t days of degradation, m_{wt} is the value of the mass of the wet swollen sample after t days.

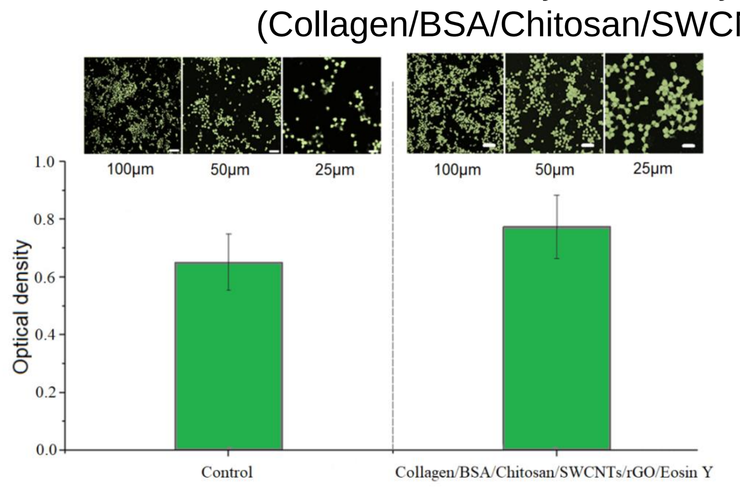


Figure 8. Image of cells after three days of observation

Conclusions

Specific conductivity: 17 mS/cm (rGO), 19 mS/cm (SWCNT) and 35 mS/cm (SWCNT/rGO). The mass loss of the SWCNT/rGO sample was 40%, the swelling increased by 20%, the optical density (OD) of the MTT test was 0.76 (Control cover glass OD=0.62).

The developed composite in the process of degradation can be replaced by biological tissue during this period with the maintenance of electrical conductivity.

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