Supplementary information

Biocompatible Electrochemical Pseudocapacitors from Graphene Oxide Nanocapsules

R.Kataky,[a*] S.Pyner,[b] F.Shenton,[b] N.Ntola,[a] M.Chowdhury,[a] A.R.Duckworth,[a] B.P.Dobson,[a] K.S.Coleman[a], and J.H.L.Hadden[a].

Fig S1 and Table S1: XRD : The X-ray diffraction patterns of GO, PVA (Si), PVA-GO, and PVA-GO (Si) films were analysed with wide angle X-ray scattering (Bruker D8) technique using Cu Kα radiation (λ= 0.154 nm). Each of the samples was run for an hour. The XRD measurements were carried out in the 2θ angle with the range of 10– 43°.

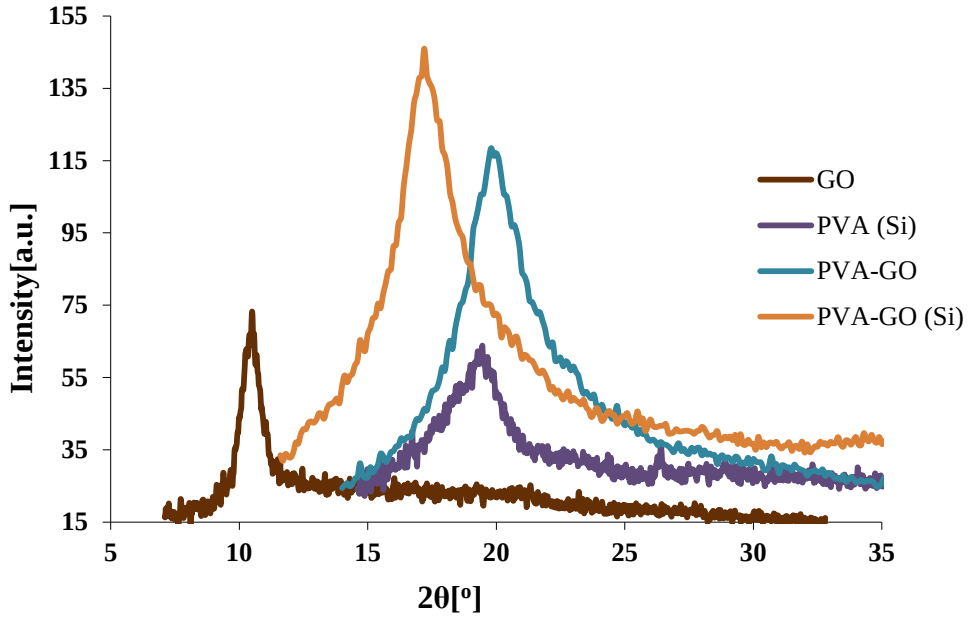


Figure S1: XRD data for GO, PVA (Si), PVA-GO, and PVA-GO (Si)

SAMPLE	2θ /deg	d / nm
GO	10.5	0.84
PVA (Si)	19.5	0.45
PVA-GO	19.9	0.45
PVA-GO (Si)	17.2	0.51

Table S1: Two theta and d-spacing values for GO, PVA (Si), PVA-GO, and PVA-GO (Si).

Fig S2: ATR-FTIR spectra of the PVA, PVA-GO, and PVA-GO (Si) films were obtained on a single-bounce Thunderdome (Spectra-Tech) accessory Nicolet Nexus Spectrometer complete with a N_2(l) cooled HgCdTe crystal detector and diamond platform. 16 scans at 1cm^{-1} resolution were collected for each of the spectra between 500 and 4000 cm^{-1}.

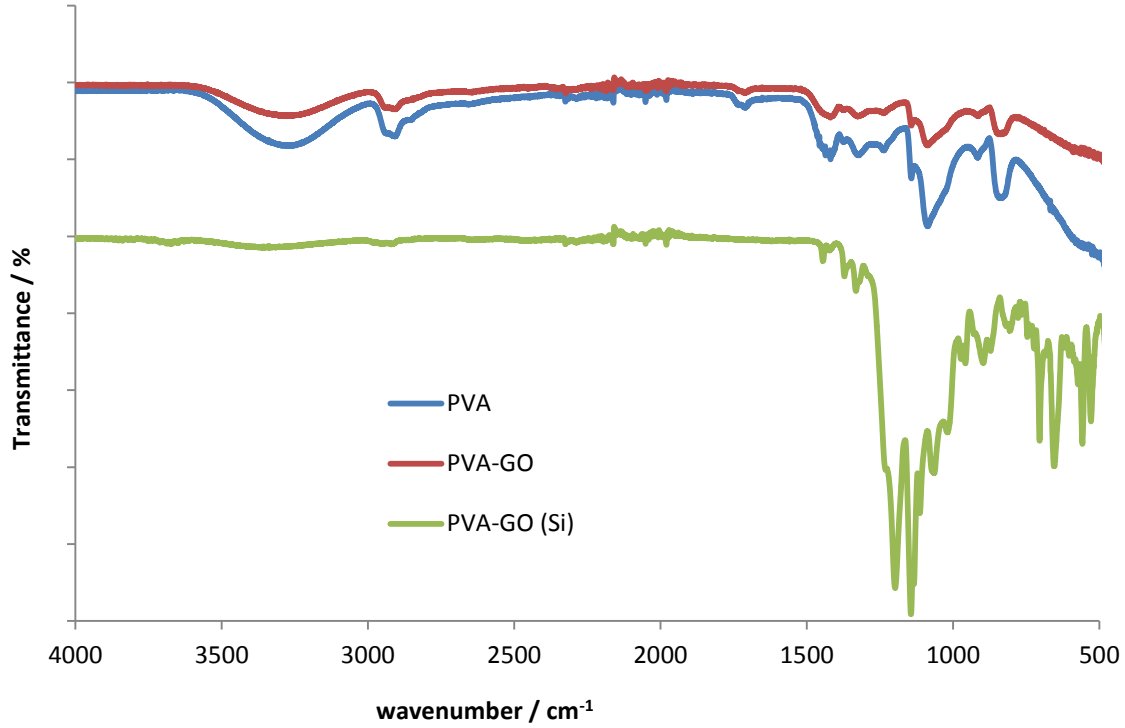


Figure S2: ATR-FTIR spectra of PVA, PVA-GO, and PVA-GO (Si).

PVA: ν_{max} / cm^{-1} 3270br (OH), 2850 (CH), 1705 (C=O), 1420 (C=C), 1090 (C-O-C)

PVA-GO: ν_{max} / cm^{-1} 3280br (OH), 2850 (CH), 1705 (C=O), 1420 (C=C), 1090 (C-O-C)

PVA-GO (Si): 1420 (C=C), 1200 (Si-O-C_xF_y), 1145 (Si-O-C/Si-O-Si), 1065 (Si-O-C/Si-O-Si), 705 (Si-C).

Fig S3: Raman spectra of film particles were collected using a Horiba Jobin Yvon LABRAM 300 system, with a 400 μm pinhole and 100 μm monochromator slit. Excitation was provided from the 632.8 nm line of a HeNe laser and delivered through a 40x long working distance confocal lens, using a 10% neutral density filter. The diameter of the confocal spot at the sample was approx. 10 μm. Back-scatter was collected through the

delivery lens and diffracted using a 600 grooves/mm diffraction grating, the spectrum being collected with a peltier-cooled Andor CCD array. The instrument was wavenumber-calibrated using the major Raman peak of a solid silicon standard (520.7 cm^{-1}). The resolution of the instrument under these conditions was 1.5 cm^{-1}.

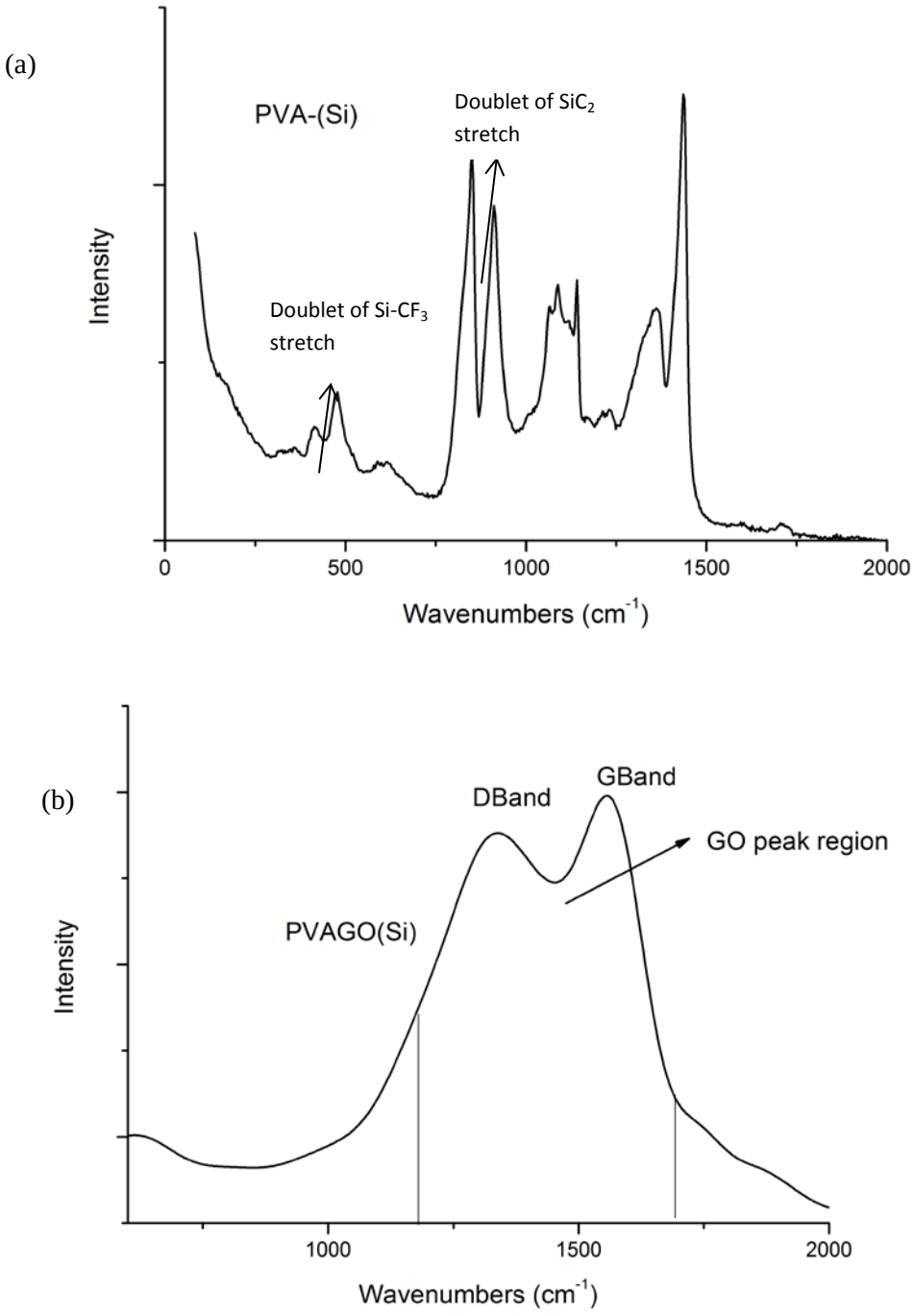
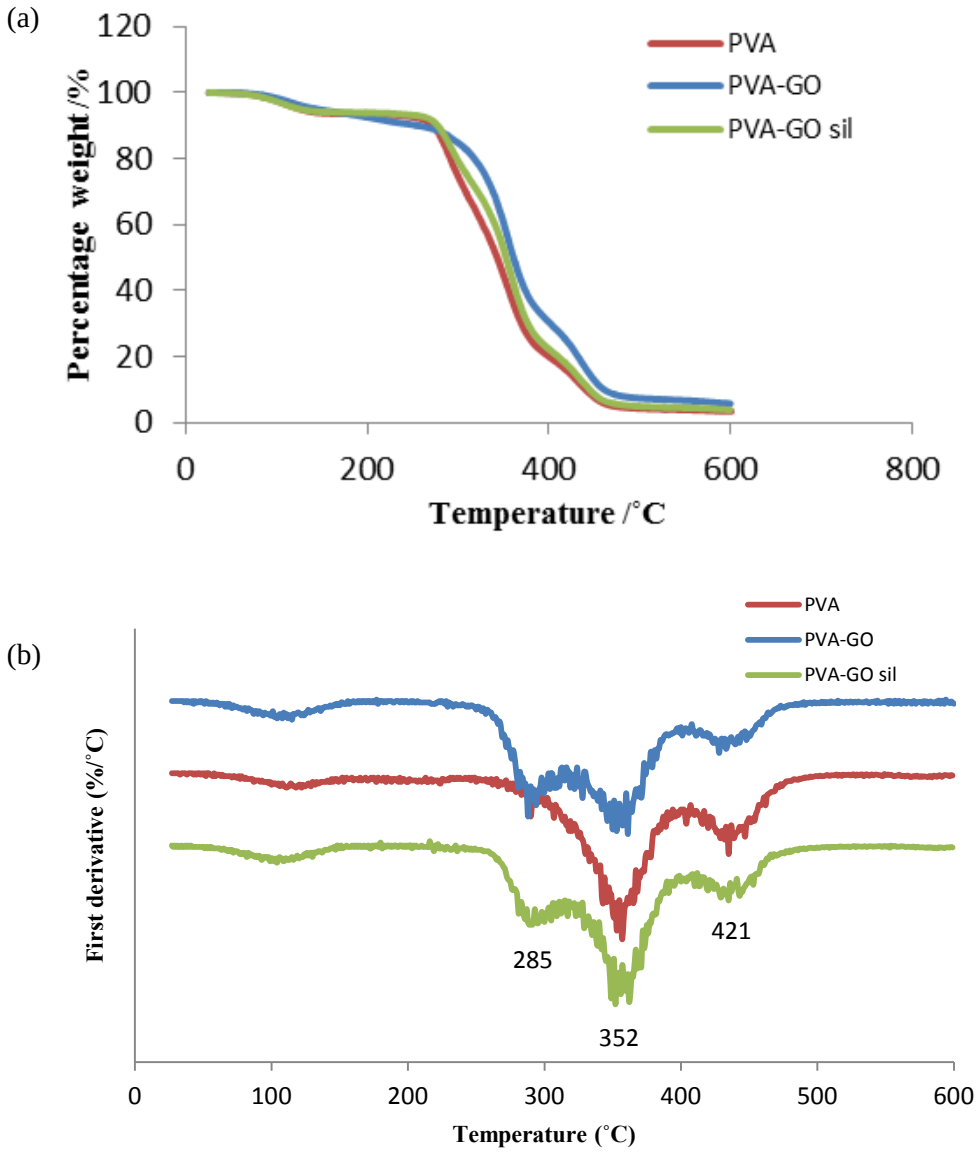


Figure S3: Raman Spectrum of (a) PVA (Si), (b) of PVA-GO (Si) composite

Fig S4: Thermal measurements; thermal transitions of the samples were investigated using Differential Scanning Calorimetry (DSC Q1000; TA instruments). The experiments were carried out in nitrogen atmosphere using approximately 7 mg sample sealed in aluminium pans. The samples were heated from room temperature to 250°C at a rate of 10°C/min.

Thermogravimetric analysis (TGA) was performed on a Perkin Elmer Pyris 1 TGA at a heating rate of 10°C/min in a nitrogen atmosphere.



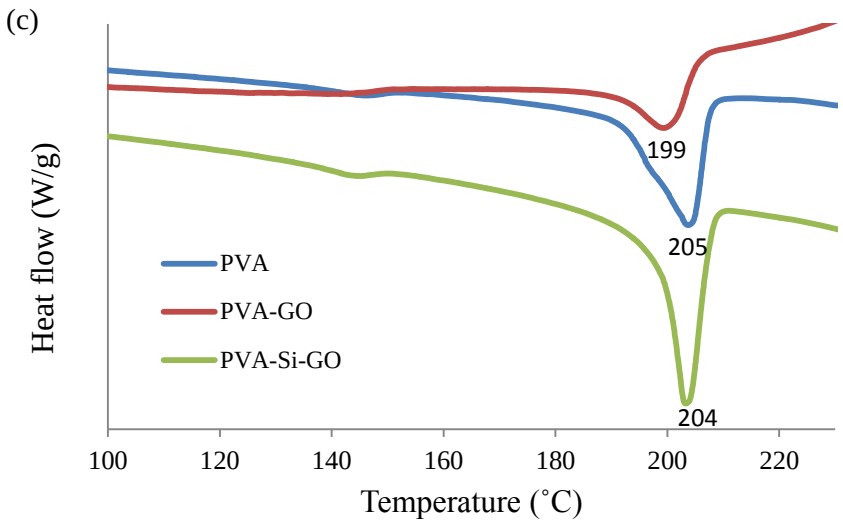


Figure S4: (a) and (b) TGA thermal stability and (c) DSC thermogram for PVA, PVA-GO and PVA-GO (Si)

Figure S5: TEM images were obtained by depositing a thin film of the PVA-GO (Si) on holey carbon grids. Low magnification TEM images and selected area electron diffraction (SAED) images were obtained using a JEOL 2100F FEG TEM with a Gatan SC-1000 Orius CCD camera at 200 kV energy. SAED images on a selected spot was obtained at 80 kV in a JEOL 2010F fitted with a CEOS aberration corrector with the spherical aberration coefficient C3 tuned to +1µm.

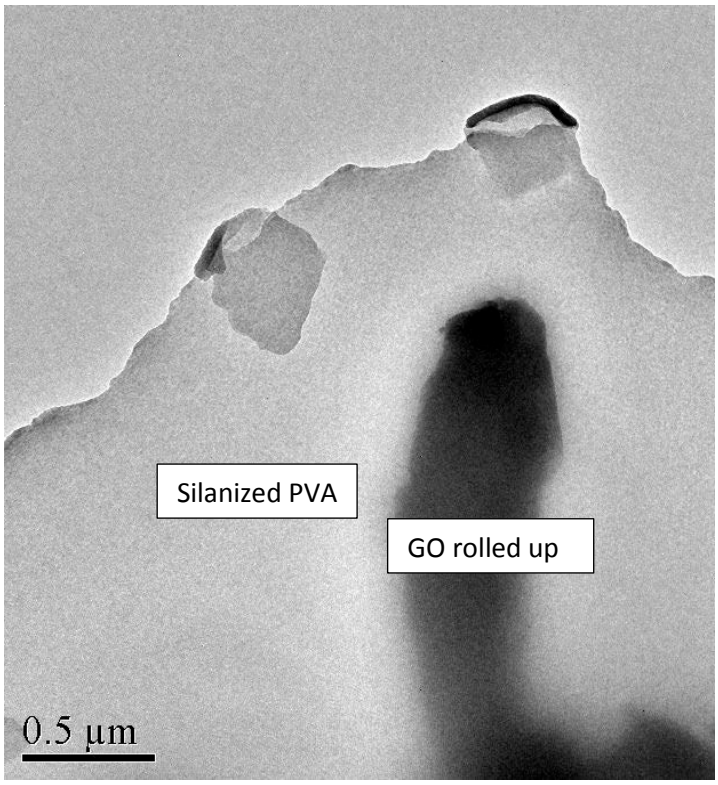


Fig S5a

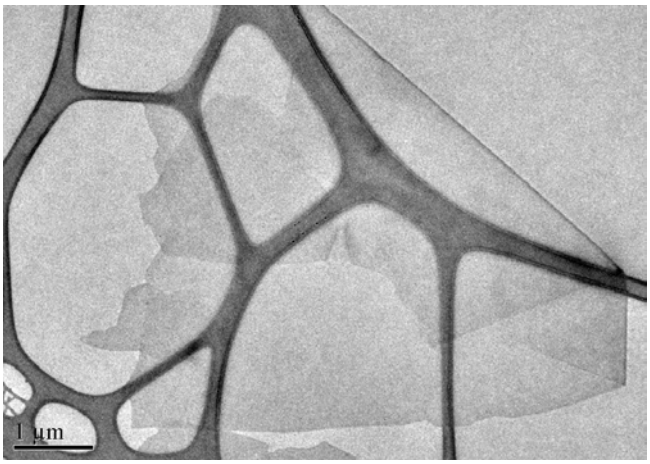


Fig S5b

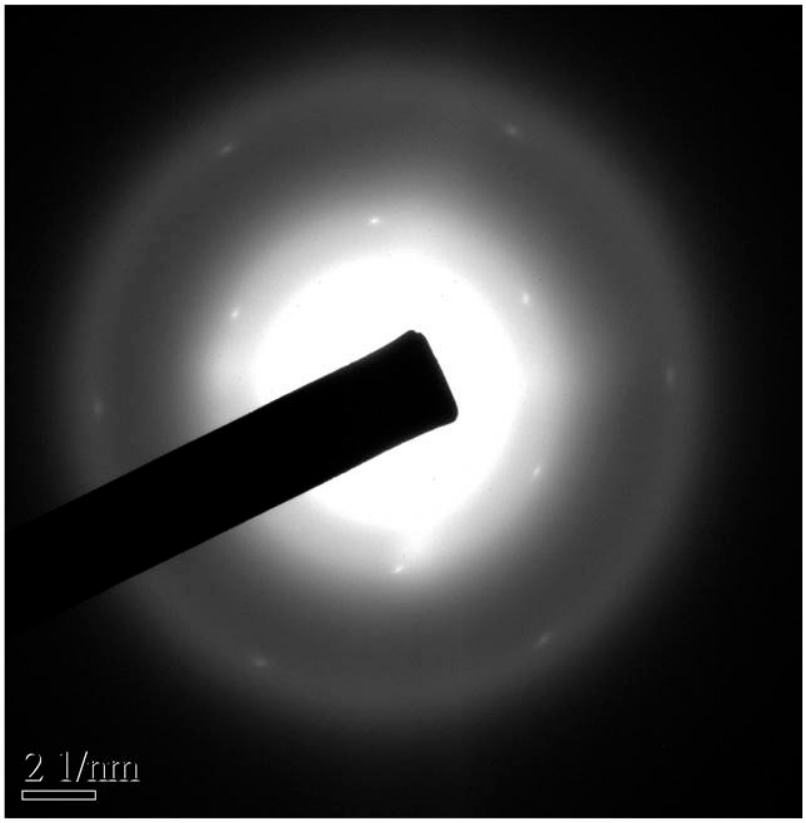


Fig S5c

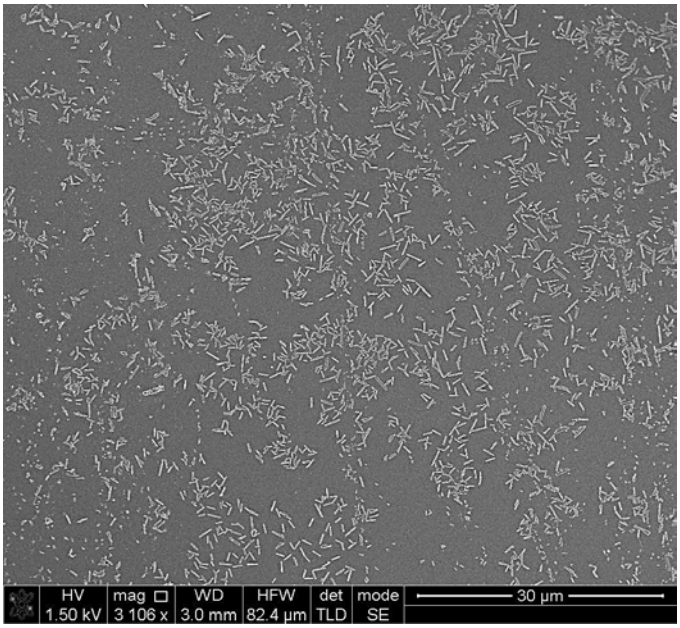


Fig S5d

Figure S5 : (a) and (b) are lower resolution TEM images of silanized PVA-GO composites, (c) electron diffraction pattern from a PVA-GO thin film and (d) a SEM image of the silanized PVA-GO composite at lower magnification.

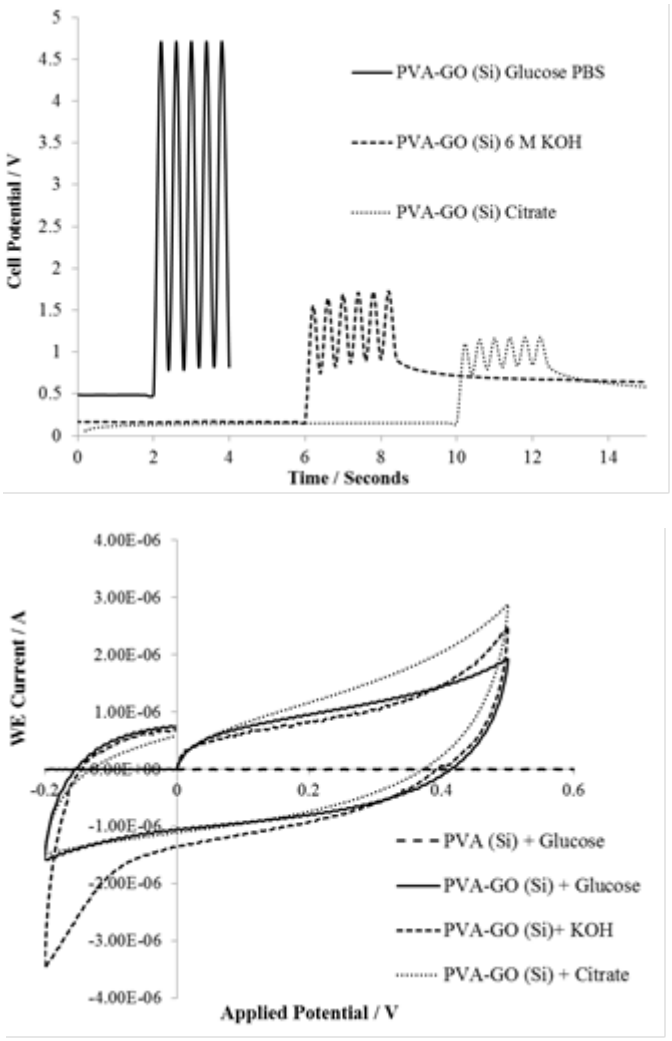


Figure S6: (a) Charge-discharge measurements (b) Cyclic voltammetry for 1:5 GO-PVA composites in a solution of phosphate buffered saline (PBS) pH 7 containing 0.50 mol dm^{-3} Glucose, citrate or aqueous 6.0 mol dm^{-3} KOH .

Figure S7: Implantation in rats: Two hydrogel composites, PVA (Si) and PVA-GO (Si), were implanted under the skin of three male rats to test for adverse reactions. Two pieces of each membrane (approximately 25 mm2) were used. Each piece of membrane was attached with Vicryl suture (W9925, Ethicon) to a short (approx. 150 mm) piece of fine bore polythene tubing (Portex tubing 800/100/200, Smiths Medical). In order to make it easier to locate and recover the membrane at the end of the experimental period, the membranes were attached to the tubing which in turn was sterilized using UV irradiation delivered in a BioRad GS Gene Linker UV Chamber running with the pre-set sterilization program. All animal experiments were carried out according to Animals (Scientific Procedures) Act 1986. Three male Wistar rats (275-315g) were anaesthetized with Isofluorane (4% delivered in oxygen at a rate of 2l/min). Using aseptic technique throughout a dorsal midline incision was made in the T5-T9 region of the spinal cord. The skin was separated from the back muscles using blunt dissection and the tubing and membrane arrangement sutured to the exposed muscle. The wound was closed and sutured using Prolene (W8871, Ethicon). Following surgery, analgesia was administered (0.01 ml/100 g buprenorphine). The rats were monitored for 7 weeks with ad libitum food and water. The rats were humanely killed by exposure to a rising concentration of CO2 gas. The skin was opened as before and peeled away to expose the implanted membranes. The area around the membranes was carefully examined and photographed using a Nikon digital camera (Coolpix 950). The membranes were removed, photographed and retained for examination under SEM.

All experiments were approved by the Local Ethics Committee of Durham University and performed in accordance with the UK Animals (Scientific Procedures Act) 1986 under a project licence granted to Dr Susan Pyner.

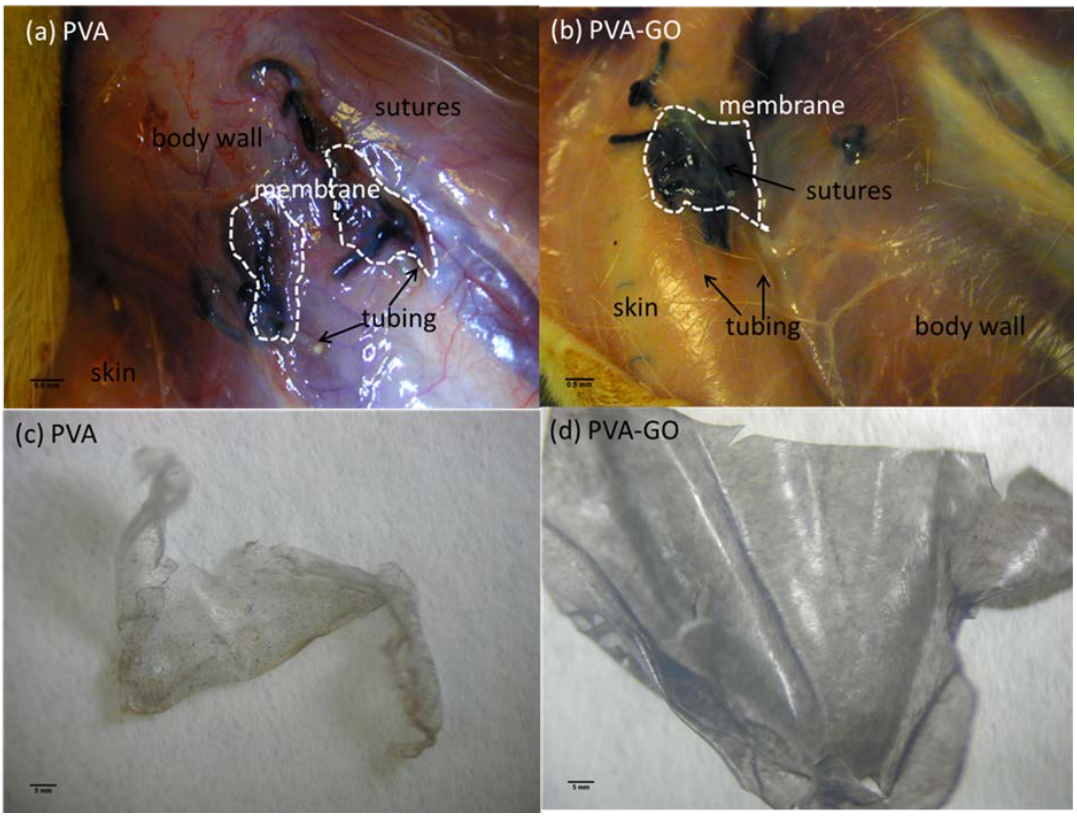


Figure S5: Silanized membranes in-situ (indicated by arrows) at seven weeks after in rabbits implantation (a) PVA (Si) (b) PVA-GO (Si). State of membranes after removal (c) PVA (Si) (d) PVA-GO (Si)