Temperature-Responsive Bioactive Glass/Polymer Hybrids Allow for Tailoring of Ion Release

Supplementary Information

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Instrumentation:

X-ray diffraction

The amorphous structure of the synthesized bioactive glass nanoparticles was analysed by X-ray diffraction (XRD) using Rigaku, MiniFlex 300, Japan. The measurement was processed in the 2θ range of 10° - 70° with a step size of 0.02° and a test speed of 1.0° per minute.

Dynamic light scattering

Dynamic light scattering (DLS) measurements were conducted using an ALV Laser CGS 3 Goniometer equipped with a 633 nm HeNe Laser. Each measurement was carried out three times at various temperatures (20-70 °C) with a detection angle of 90°.

Inductively coupled plasma optical emission spectroscopy (ICP-OES)

The concentration of different ions was measured using an Agilent 5800 inductively coupled plasma optical emission spectroscopy (ICP-OES) instrument. Standard solutions used for calibration were prepared by diluting single-element ICP standard solutions obtained from Carl Roth (1000 mg/L). The dilution process involved preparing six solutions with concentrations ranging from 0.001 mg/mL to 10 mg/L. Additionally, 28% nitric acid (HNO_3) was used for the dilution. As the Ca^{2+} concentration in samples was outside the preferable measurement range, 1 mL of sample was diluted to 10 mL. 0.5 mL of concentrated HNO3 was added to keep samples in an acidic condition. Triplicate measurements were taken for each sample during measurements. The ion concentrations without dilution were recalculated after the measurement.

Ultraviolet-visible spectroscopy

UV/vis measurements were conducted using an Agilent Cary 60 spectrophotometer in Hellma quartz glass cuvettes with a path length of 10 mm. Each sample was dissolved in Milli-Q water at room temperature and measured three times independently using fresh dispersion solutions. The solutions were transferred to quartz cuvettes and heated gradually from 25 to 70 °C at a rate of 0.2 °C/min without agitation. UV/vis spectroscopy was utilized to collect turbidity data by measuring the absorbance at λ = 500 nm until 50% transmittance was achieved. The transition window (ΔT), with an error < 0.1°C per minute, representing the temperature range between 1% and 99% transmittance, was then determined.

Scanning electron microscopy

Scanning electron microscopy (SEM) images were captured using a Zeiss SIGMA VP Field Emission SEM equipped with a GEMINI column from Carl-Zeiss AG, Germany. The SEM operated at 3–7 kV and employed either the InLens or SE2 detector. Samples were directly collected from the dispersion solution in EtOH and subsequently loaded onto carbon-coated stubs. Coating was performed using sputtering techniques with either Pt or Au.

Transmission electron microscopy

For transmission electron microscopy (TEM) from aqueous solutions, copper grids were made hydrophilic by Ar plasma cleaning for 120 seconds (Diener Electronics). Subsequently, 10 µL of the respective sample solution was applied to the grid, and any excess sample was carefully blotted with filter paper. TEM images were captured using a 200 kV FEI Tecnai G2 20 microscope equipped with a 1k x 1k Olympus MegaView camera for image acquisition.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) measurements were conducted in a PerkinElmer TGA8000 under a nitrogen atmosphere (20 mL min^{-1}), covering a temperature range from 30 °C up to 850 °C. The heating rate employed for the analysis was 10 K min^{-1}.

FT-IR measurements

Infrared spectra were obtained using a PerkinElmer Frontier Fourier transform infrared (FT-IR)/NIR spectrometer, which was equipped with a Golden Gate ATR unit from Specac. The spectra were recorded within the range of 4000 to 400 cm^{-1}.

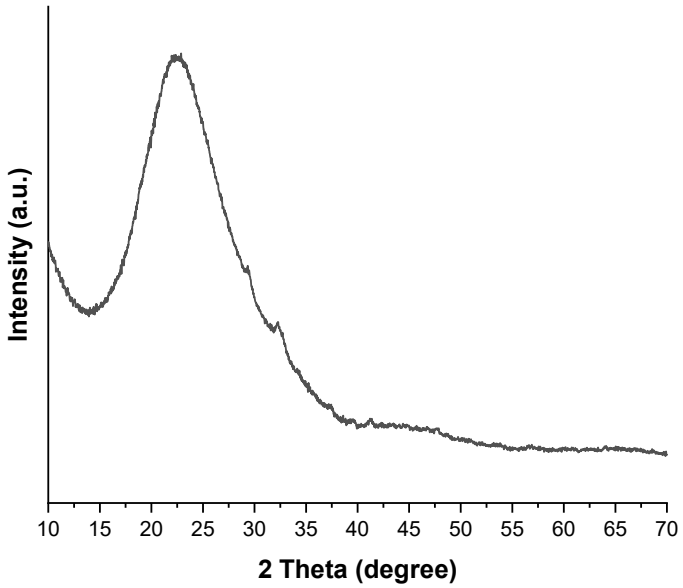

Figure S1: X-ray diffraction pattern of BG nanoparticles Si85-Ca15

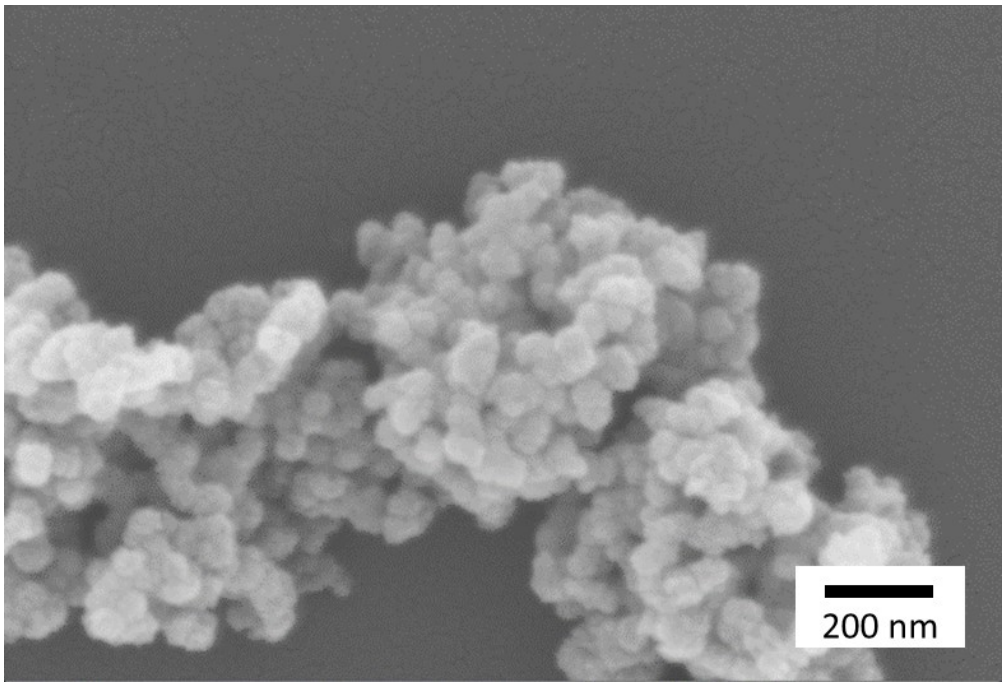

Figure S2: SEM image of BG nanoparticles

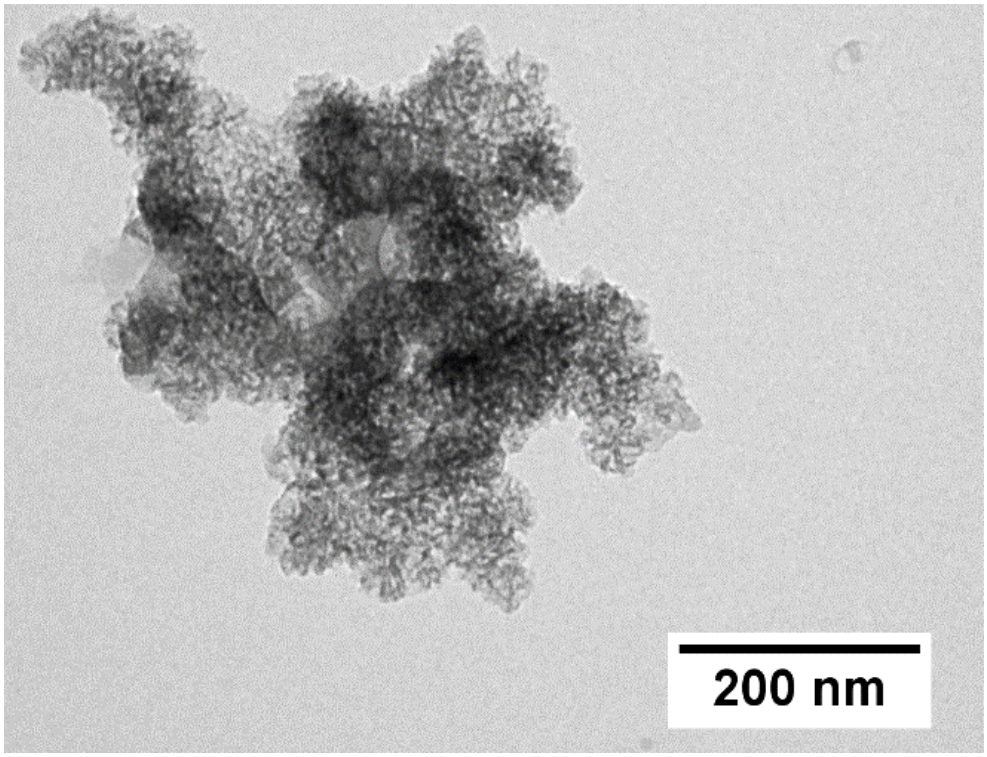

Figure S3: TEM image of BG nanoparticles

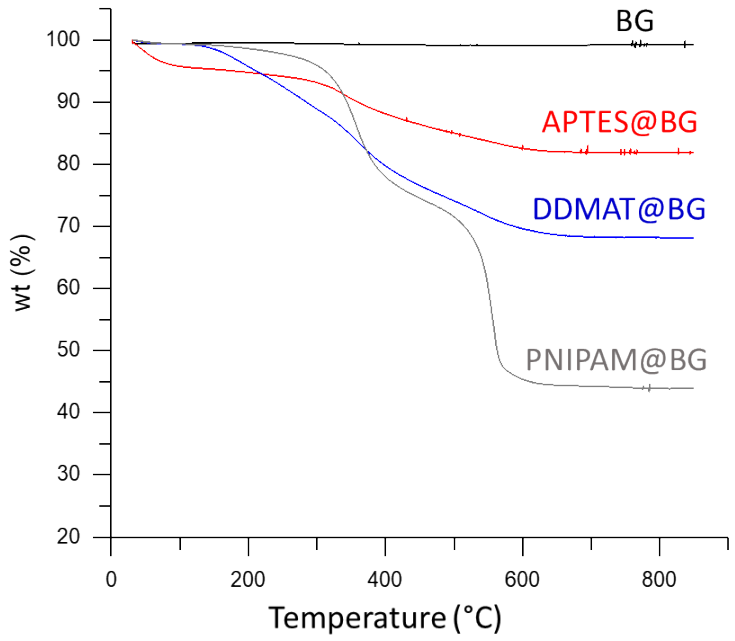

Figure S3: Thermogravimetric analysis of BG particles, APTES@BG, DDMAT@BG, and PNIPAM@BG nanoparticles.

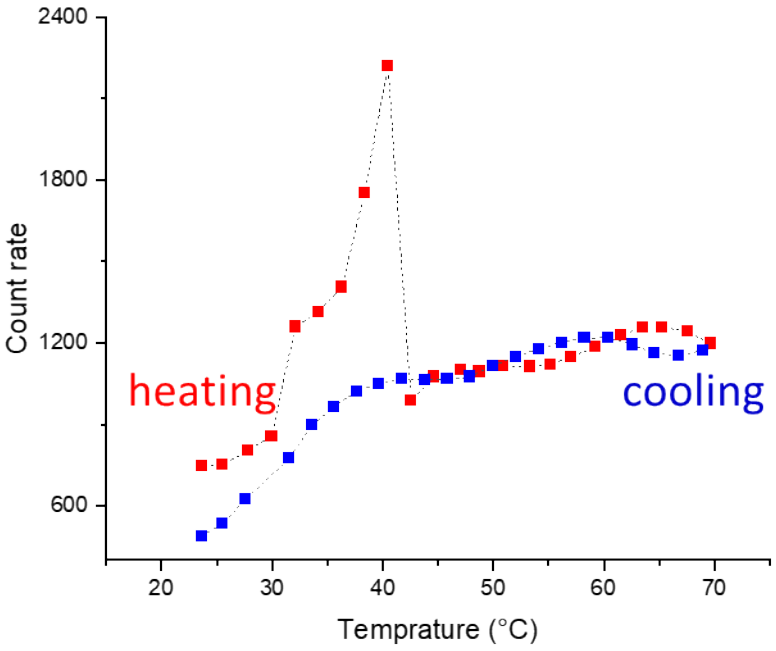

Figure S4: Normalized scattering intensity over temperature (from 25 to 70°C) for PNIPAM@BGs (1 mg/mL).