Hydrophobic bioadhesive composites for human motion detection

Singh, Manisha; Solic, Ivan; Steele, Terry W. J.

2021

Singh, M., Solic, I. & Steele, T. W. J. (2021). Hydrophobic bioadhesive composites for human motion detection. ACS Macro Letters, 10(11), 1353-1358. https://dx.doi.org/10.1021/acsmacrolett.1c00559

https://hdl.handle.net/10356/158570

https://doi.org/10.1021/acsmacrolett.1c00559

Supplementary Information

Hydrophobic bioadhesive composites for human motion detection

Manisha Singh[1,2], Ivan Solic[2], Terry W. J. Steele[1,2*]

[1]NTU-Northwestern Institute for Nanomedicine (NNIN), Interdisciplinary Graduate School (IGS), Nanyang Technological University (NTU), Singapore 637553

[2]School of Materials Science and Engineering (MSE), Nanyang Technological University (NTU), Singapore 639798

[*]Correspondence to Terry W. J. Steele (e-mail: wjsteele@ntu.edu.sg)

CaproGlu Synthesis

4-[3-(Trifluoromethyl)-3H-diazirin-3-yl] benzoic acid (230.14 $g.mol^{-1}$ Dz-COOH) was synthesized from Dz-CH2OH. The solution of Dz-CH2OH (1 mol. eq.) and potassium hydroxide (1.55 mol. eq.; 0.6 M KOH solution) was dissolved in a mixture of water/dioxane (5/1), cooled down to 0 °C in an ice bath and potassium permanganate (1.5 eq.) was added portion-wise. The biphasic reaction mixture was vigorously stirred at room temperature overnight (18 hours). The suspension was filtered through a pad of Celite®. The filtrate was cooled in an ice bath and acidified with 2M HCl to pH=1. The obtained white precipitate was filtered off and dissolved in ethanol. The organic layer was washed 5 times with water and one time with brine, dried with anhydrous MgSO4, and the solvent was evaporated in a vacuum to yield pure Dz-COOH as a pale-yellow solid. The Proprietary CaproGlu synthesis method was the esterification reaction between PCLT (polyol) and Dz-COOH (acid) with 1,1-carbonyldiimidazole (CDI) used as a coupling agent. The molar ratio of Dz-COOH/PCLT = 2/1 was deliberately chosen to yield ~50% diazirine conjugation. The reactants were placed in a round-bottomed flask with a magnetic stir bar. The flask was immersed in a water bath placed on the top of the heater equipped with a magnetic stirrer. DCM was added dropwise and stirred at room temperature for 1 h. The solution of PCLT in DCM was added into the reaction mixture slowly and the round bottom flask was fitted with a condenser. The reaction mixture was heated to 40 °C and stirred for 18 h. After completion DCM was removed on rotavap and the product CaproGlu was purified by water / Et_2O extraction (the water phase contained saturated $NaHCO_3$ to remove unreacted Dz-COOH). The organic layer was dried with anhydrous $MgSO_4$

and filtrated. Et_2O was removed with rotavap. Traces of solvent from CaproGlu product were removed on a high vacuum for 6 h at 40 °C.

Technical details on the CNTs, as provided by the manufacturer [1]:

Properties of CNT	**Value**
Average diameter	9.5×10^{-9} m
Average length	1.5 µm
Carbon purity	90 %
Surface Area	250-300 m^2/g
Volume resistivity	10^{-4} Ω cm
[1]NC7000™ - Technical Data Sheet (DM-TI-02-TDS-NC7000-V08). https://www.nanocyl.com/download/tds-nc7000/	

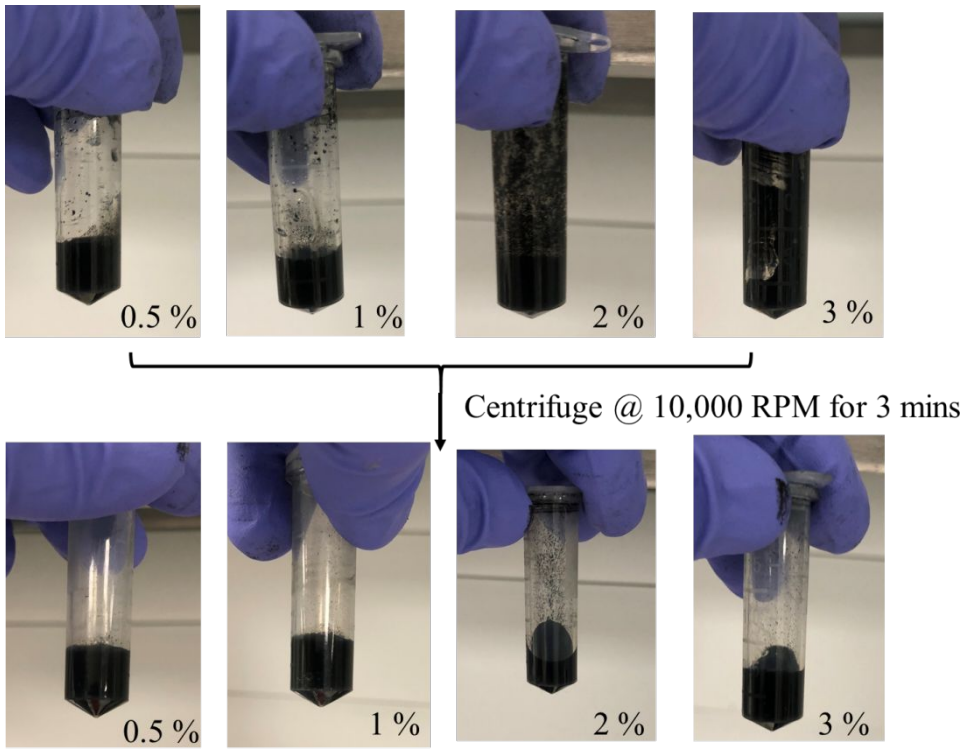

Figure S1: CNTs were physically mixed and remained dispersed in CaproGlu even after centrifuging at 10000 RPM for 3 minutes.

Scanning Electron Microscopy (SEM)

For qualitative analysis of CNTs dispersion in the UV-cured CaproGlu matrix, SEM analyzed the morphology of the composites. Cured samples were subjected to platinum coating (90 s, chamber pressure <5 Pa at 20 mA). Images were obtained at an acceleration voltage of 5 kV and a working distance of 8-9 mm (**Figure S4**).

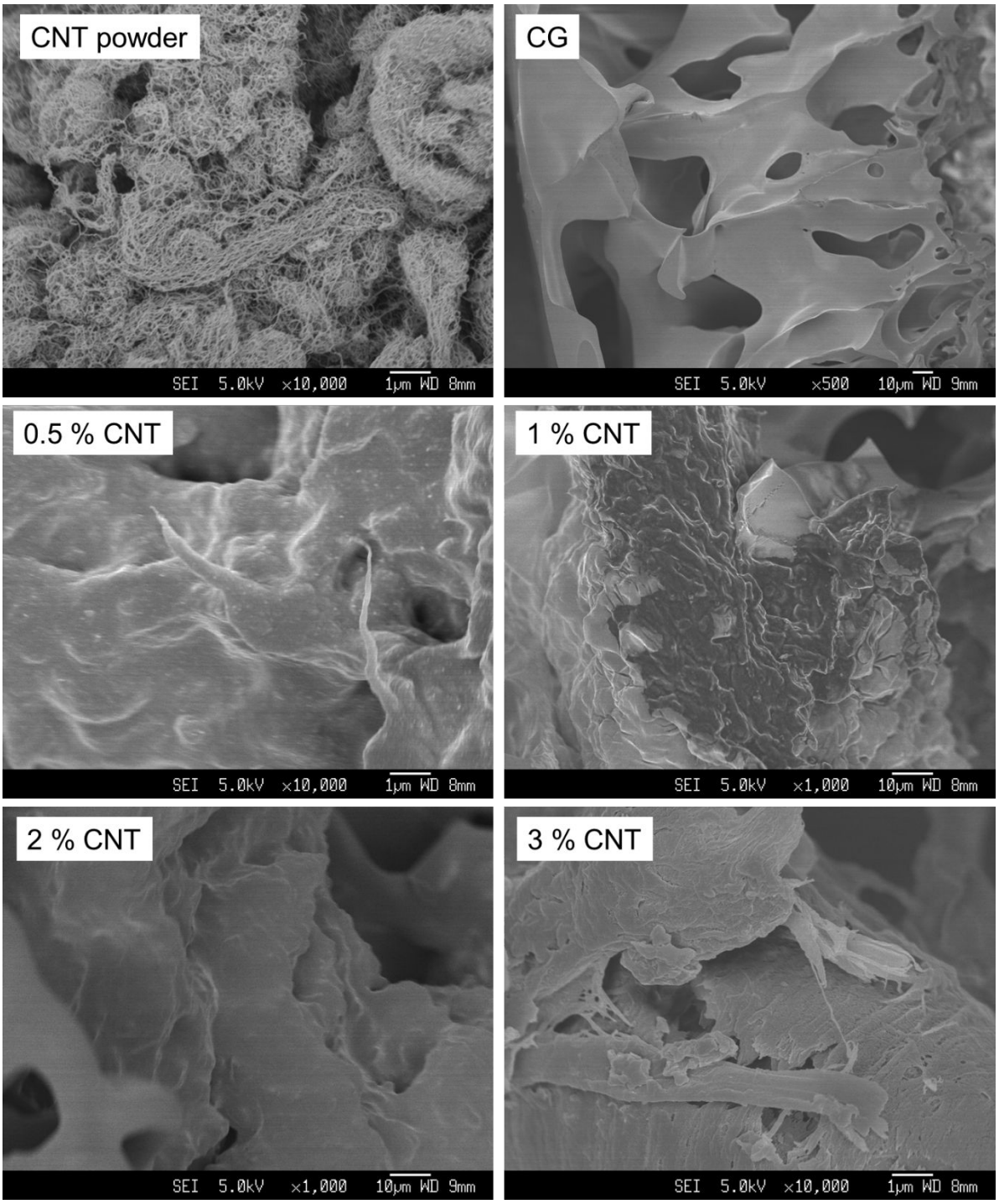

Figure S2: Cross-sectional morphology of CaproGlu composites under SEM. The surface of CaproGlu control (CG) seems smooth, whereas the cross-sectional morphology of the composites shows rough-textured dispersion of CNTs in the matrix.

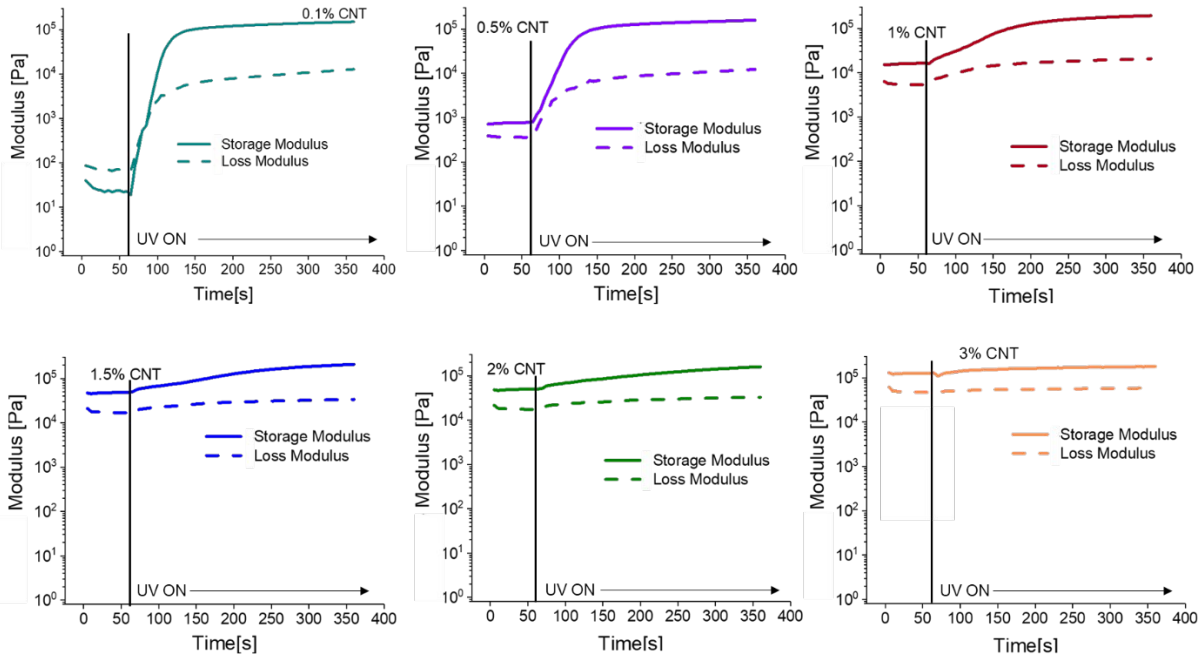

Figure S3: Real-time stimuli-based oscillatory rheology for CaproGlu composites showing G' and G".

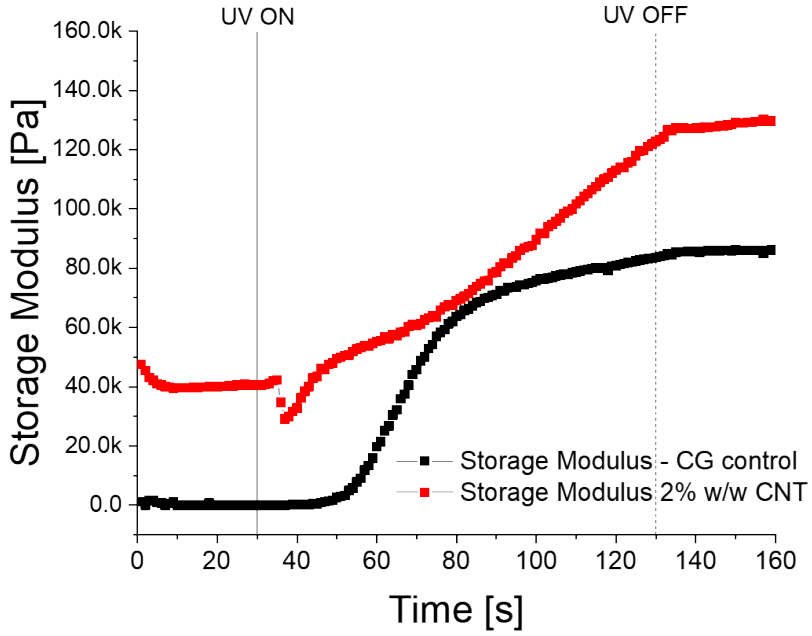

Figure S4: Real-time stimuli-based oscillatory rheology for CaproGlu composites showing G' stabilizes once UV is turned off.

Mechanical, adhesion, and electrical properties of the composites under thermal curing at 105 °C

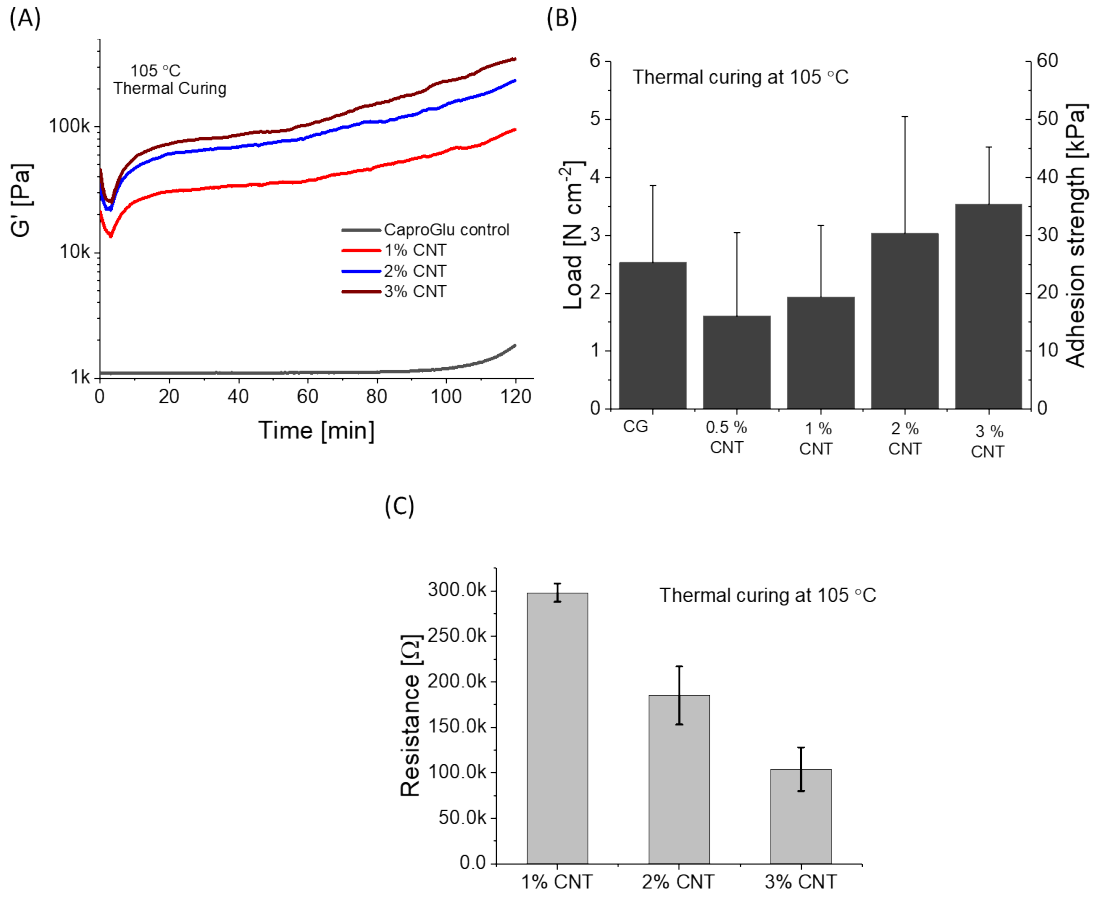

Figure S5: Evaluation of CaproGlu composites under thermal curing at 105 °C. (A) Comparison of real-time stimuli-based oscillatory rheology (G') for CaproGlu composites. (B) Comparison of maximum lap-shear adhesion strength at a strain rate of 3 mm min^{-1}. (C) Comparison of resistance measured by the two-point probe method.