Supporting Information

Boron Nitride Nanotubes Enhance Mechanical Properties of Fibers from Nanotube/Polyvinyl Alcohol Dispersions

*Joe F. Khoury, Jacob C. Vitale, Tanner L. Larson, Geyou Ao**

Department of Chemical and Biomedical Engineering, Washkewicz College of Engineering, Cleveland State University, 2121 Euclid Avenue, Cleveland, OH 44115, USA

Corresponding Author

* g.ao@csuohio.edu

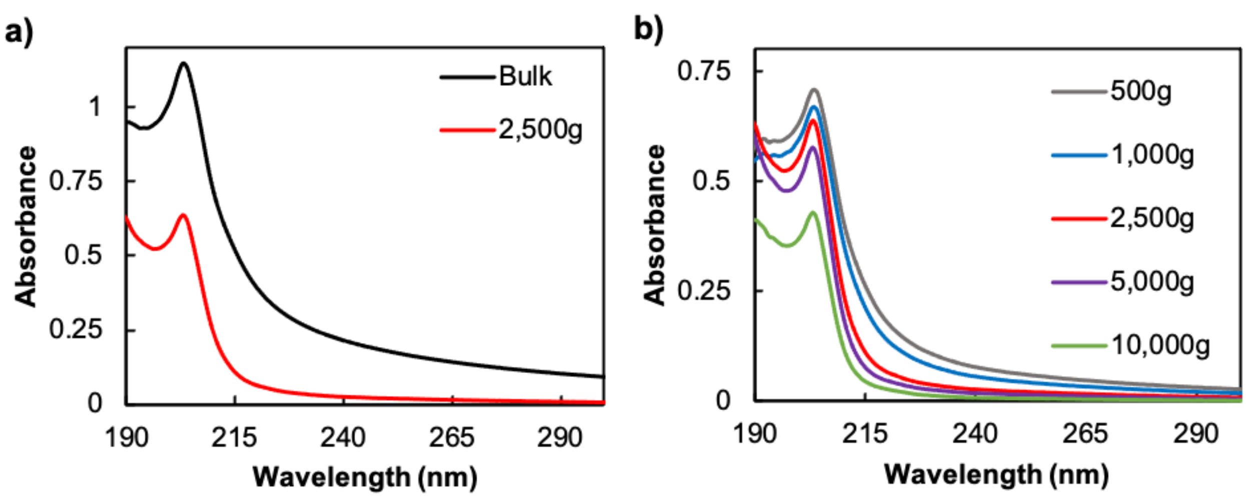

Figure S1. Absorbance spectra of (a) bulk (i.e., without centrifugation) and supernatant (i.e., after centrifugation at 2,500g) dispersions as well as (b) supernatant dispersions collected after varying centrifugation at 500-10,000g. The starting sample is 1 mg/mL pristine BNNTs-1 mass % SDC dispersion in water. Samples were diluted by a factor of 200× in DI water for UV-vis absorbance measurements.

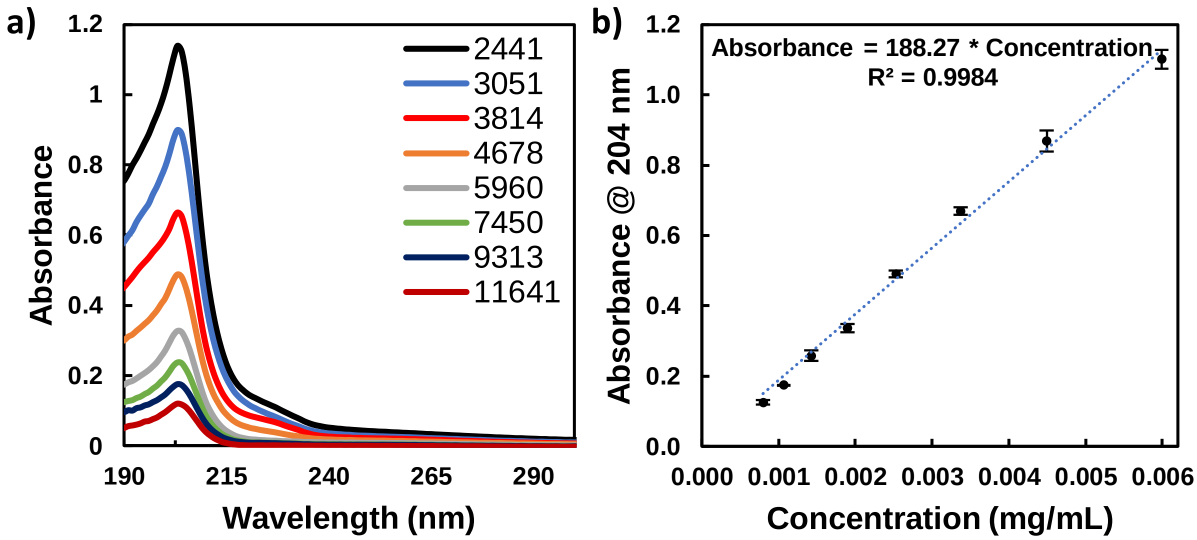

Figure S2. Determining the extinction coefficient of SDC-BNNT complexes at 204 nm. (a) Absorbance spectra of a concentrated, supernatant dispersions of pristine BNNTs stabilized by SDC at different dilution factors ranging from 2441× to 11641× in DI water. (b) The calibration curve of SDC-BNNT absorbance values obtained at 204 nm. The extinction coefficient is determined to be 188.27 mL mg^{-1} cm^{-1} at 204 nm. Error bars were generated from the standard deviation of three repeats.

Table S1. Zeta potentials measured for surfactant-dispersed purified BNNTs in water.

Surfactant-coated BNNTs	Zeta potential (mV)
SDC-BNNTs	-53 ± 0.83
SDS-BNNTs	-57 ± 0.16
TTAB-BNNTs	+52 ± 0.41

Table S2. Estimated BNNT contents in BNNTs/PVA fibers based on the compositions of BNNTs/PVA dispersions.

BNNTs/PVA Dispersions			**BNNTs/PVA Fibers**	
BNNTs (mass %)	**SDC (mass %)**	**PVA (mass %)**	**BNNTs (mass %)**	**BNNTs (vol %)**
0.025	0.12	5.00	0.49	0.45
0.050	0.24	5.00	0.94	0.88
0.075	0.36	5.00	1.38	1.27
0.010	0.47	5.00	1.79	1.64

*****Conversion of nanotube mass % to vol % is provided for fibers produced from purified BNNTs in 5 mass % PVA solution. BNNT content in dispersions was assumed to be maintained in the resulting BNNTs/PVA fibers.

Table S3. Mechanical properties of neat PVA, pristine BNNTs/PVA, and purified BNNTs/PVA fibers produced from dispersions containing 5 mass % PVA in an EtOH coagulation bath.

Fibers	**BNNTs in dispersion (mass %)**	**Tensile strength (MPa)**	**Young's modulus (GPa)**	**Toughness (J/g)**	**Strain at failure (%)**
Neat PVA	0	185 ± 45	1.1 ± 0.1	112 ± 31	88 ± 40
Pristine BNNTs/PVA	0.025	274 ± 65	2.1 ± 0.3	112 ± 15	131 ± 31
	0.050	297 ± 46	2.1 ± 0.4	137 ± 11	96 ± 19
	0.075	329 ± 18	5.4 ± 0.9	99 ± 24	65 ± 25
	0.100	445 ± 21	6.8 ± 1.2	135 ± 19	69 ± 13
Purified BNNTs/PVA	0.025	307 ± 62	3.8 ± 0.5	102 ± 39	95 ± 49
	0.050	411 ± 37	3.5 ± 0.2	141 ± 21	91 ± 23
	0.075	360 ± 3	7.1 ± 1.0	150 ± 57	55 ± 7
	0.100	553 ± 45	7.1 ± 0.8	166 ± 55	65 ± 25

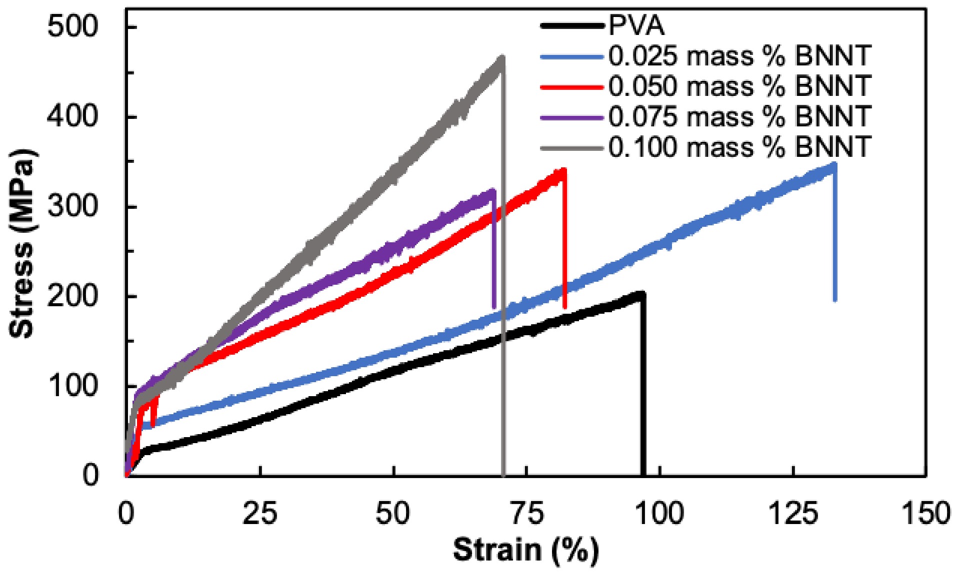

Figure S3. Representative stress-strain curves of neat PVA and pristine BNNTs/PVA fibers produced from dispersions with increasing nanotubes concentrations from 0.025 to 0.100 mass % and a constant PVA concentration of 5 mass %. The fibers were produced in an EtOH bath.

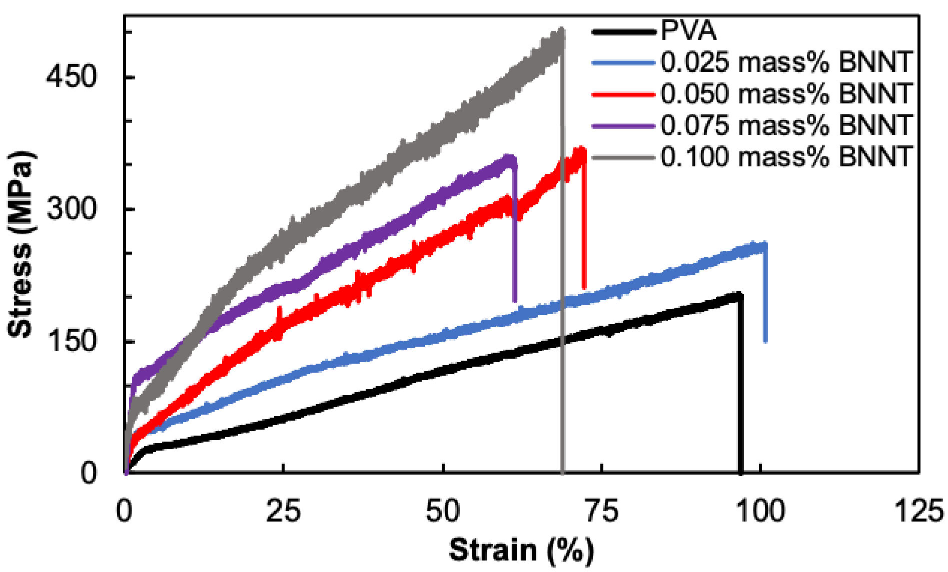

Figure S4. Representative stress-strain curves of neat PVA and purified BNNTs/PVA fibers produced from dispersions with increasing nanotube concentrations from 0.025 to 0.100 mass % and a constant PVA concentration of 5 mass %. The fibers were produced in an EtOH bath.

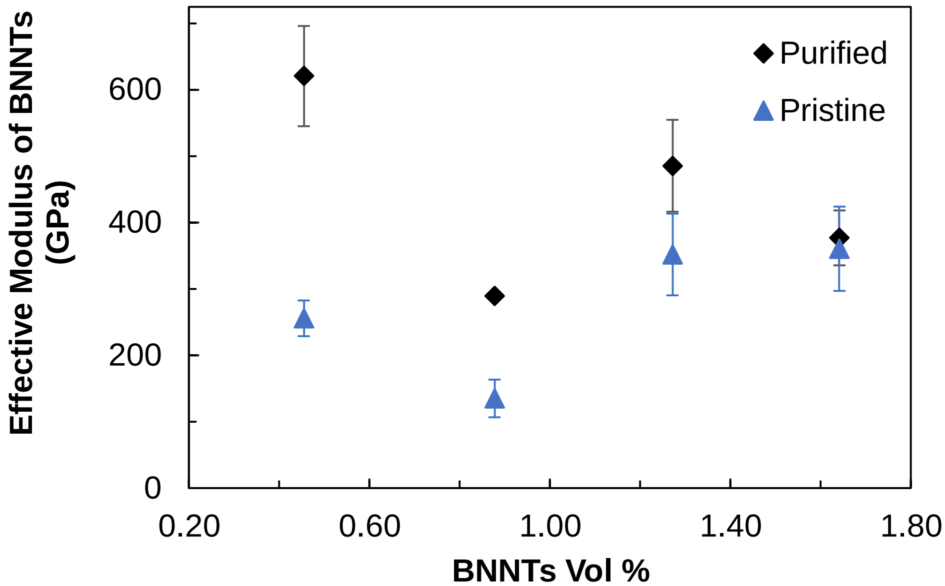

Figure S5. Estimated effective modulus of pristine and purified BNNTs as a function of BNNTs vol % in BNNTs/PVA fibers produced from dispersions with a constant PVA concentration of 5 mass %. The fibers were produced in an EtOH bath.

The effective moduli of BNNTs in BNNTs/PVA fibers are estimated based on the rule of mixture[1,2] utilizing $E_{BNNTs/PVA} = V_{BNNTs}E_{BNNTs} + V_{PVA}E_{PVA}$, where $E_{BNNTs/PVA}$, E_{BNNTs}, E_{PVA} are the Young's modulus of BNNTs/PVA fibers, BNNTs, and neat PVA fibers, respectively, and V_{BNNTs} and V_{PVA} are the volume fractions of BNNTs and PVA in fibers, respectively.

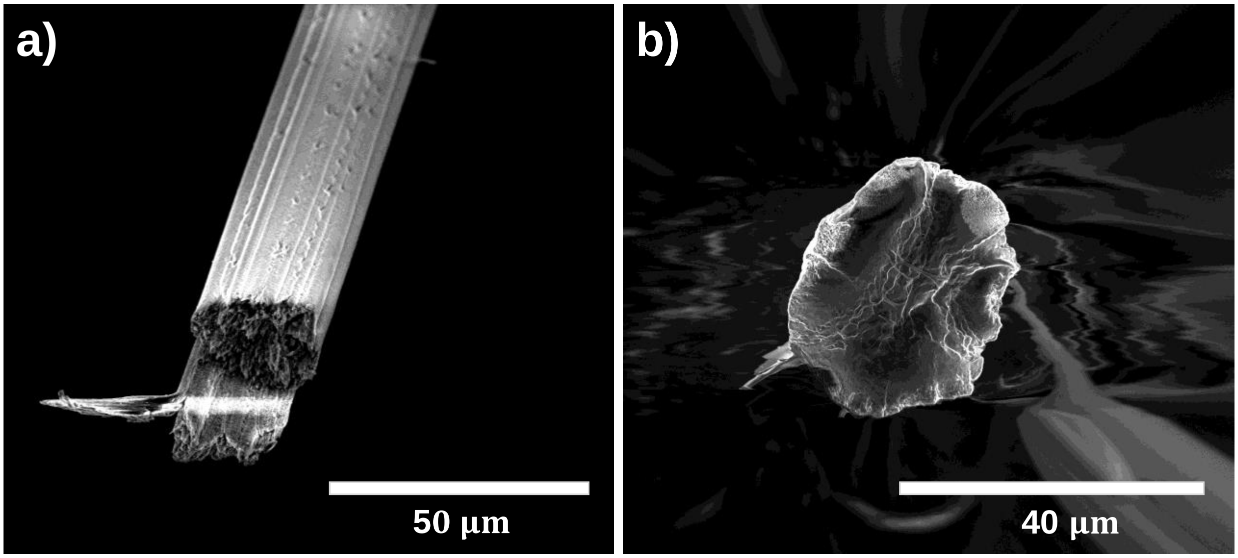

Figure S6. SEM image showing the cross section of (a) pristine BNNTs/PVA composite fiber produced from an aqueous solution of 0.025 mass % BNNTs and 5 mass % PVA in EtOH coagulation bath and (b) PVA only fiber produced from an aqueous solution of 5 mass % PVA in an MeOH coagulation bath.

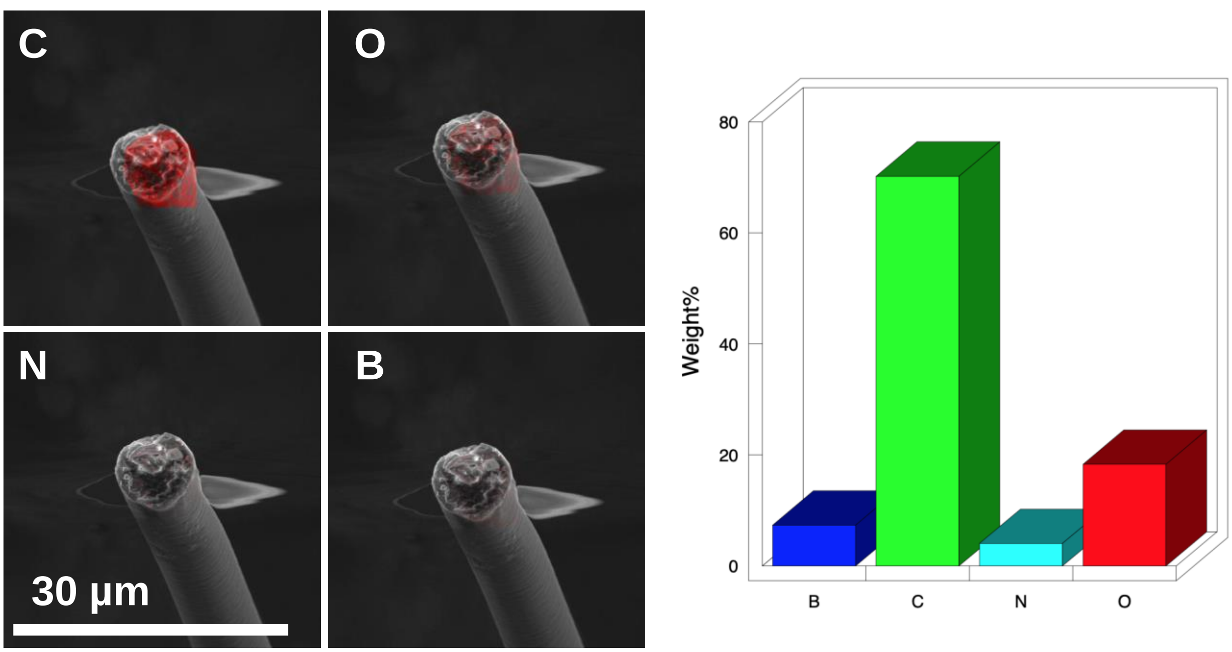

Figure S7. Elemental mapping of the cross section of the purified BNNTs/PVA fibers produced from an aqueous solution of 0.1 mass % BNNTs and 2.5 mass % PVA in EtOH coagulation bath.

Table S4. Mechanical properties of pristine BNNTs/PVA fibers produced from dispersions containing 0.1 mass % BNNTs and different PVA concentrations of 2.5 and 5 mass %, respectively, in an EtOH coagulation bath.

PVA concentration (mass %)	BNNTs in dispersion (mass %)	Tensile strength (MPa)	Young's modulus (GPa)	Toughness (J/g)	Strain at failure (%)
2.5	0.1	757 ± 147	14.0 ± 3.4	112 ± 30	60 ± 23
5.0	0.1	445 ± 21	6.8 ± 1.2	135 ± 19	69 ± 13

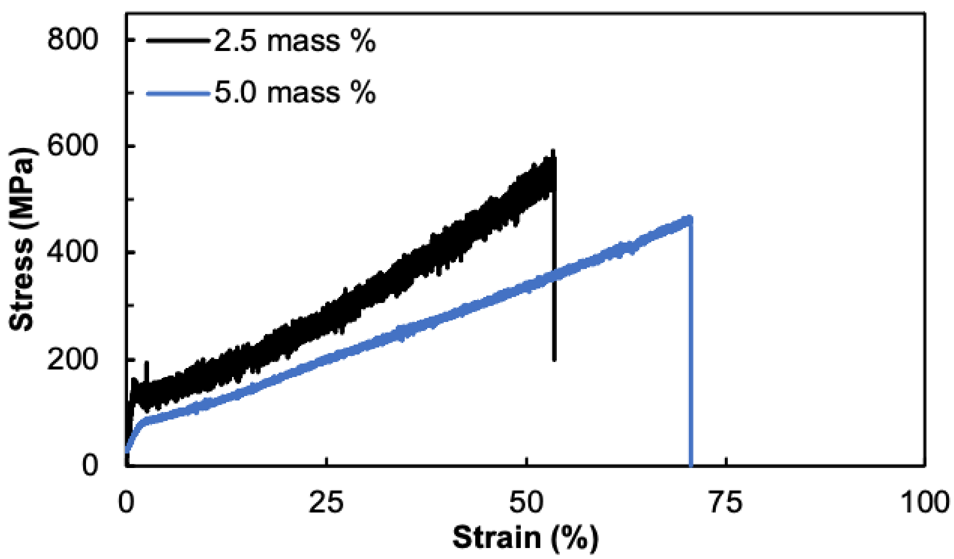

Figure S8. Representative stress-strain curves of pristine BNNTs/PVA fibers produced from dispersions containing 0.1 mass% pristine BNNTs and different PVA concentrations of 2.5 and 5 mass %, respectively, in an EtOH coagulation bath.

Table S5. Mechanical properties of pristine BNNTs/PVA fibers produced from dispersions containing 0.1 mass % BNNTs and 2.5 mass % PVA in different coagulation baths including MeOH, EtOH, and MeOH/acetone cosolvent containing 25 vol % acetone.

Coagulation bath	Tensile strength (MPa)	Young's modulus (GPa)	Toughness (J/g)	Strain at failure (%)
Ethanol	757 ± 147	14.0 ± 3.4	112 ± 30	60 ± 23
Methanol	789 ± 46	9.7 ± 3.5	140 ± 65	47 ± 19
Methanol/Acetone	313 ± 51	7.2 ± 1.5	88 ± 17	50 ± 13

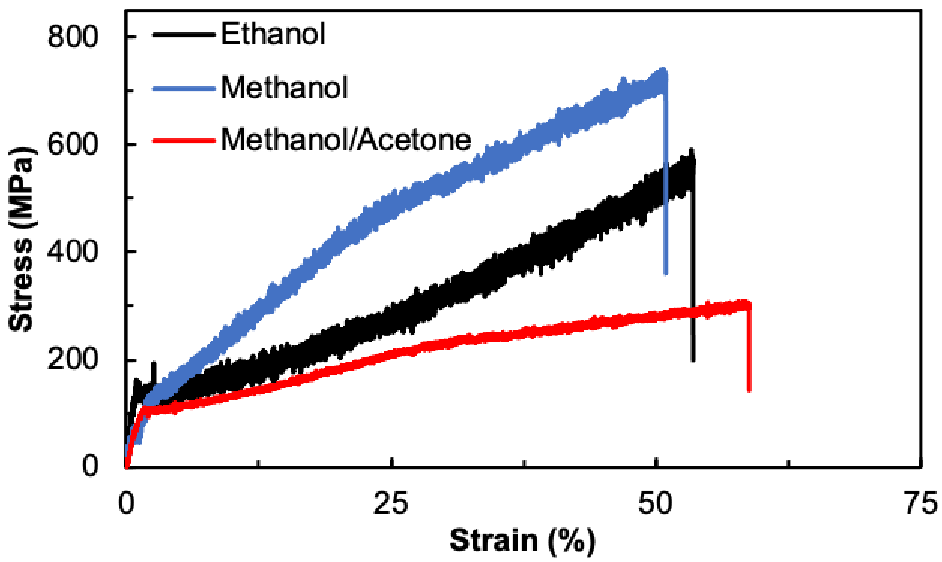

Figure S9. Representative stress–strain curves of pristine BNNTs/PVA fibers produced from dispersions containing 0.1 mass % pristine BNNTs and 2.5 mass % PVA in different coagulation baths including MeOH, EtOH, and MeOH/acetone cosolvent of 25 vol % acetone.

References:

(1) You, Y.-J.; Kim, J.-H.; Park, K.-T.; Seo, D.-W.; Lee, T.-H. Modification of Rule of Mixtures for Tensile Strength Estimation of Circular GFRP Rebars. *Polymers* **2017**, *9*, 682.

(2) Mora, R. J.; Vilatela, J. J.; Windle, A. H. Properties of Composites of Carbon Nanotube Fibers. *Comp. Sci. Technol.* **2009**, *69*, 1558-1563.