Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Cellulose nanofibrils reinforced silica aerogels: Optimization of preparation process evaluated by response surface methodology

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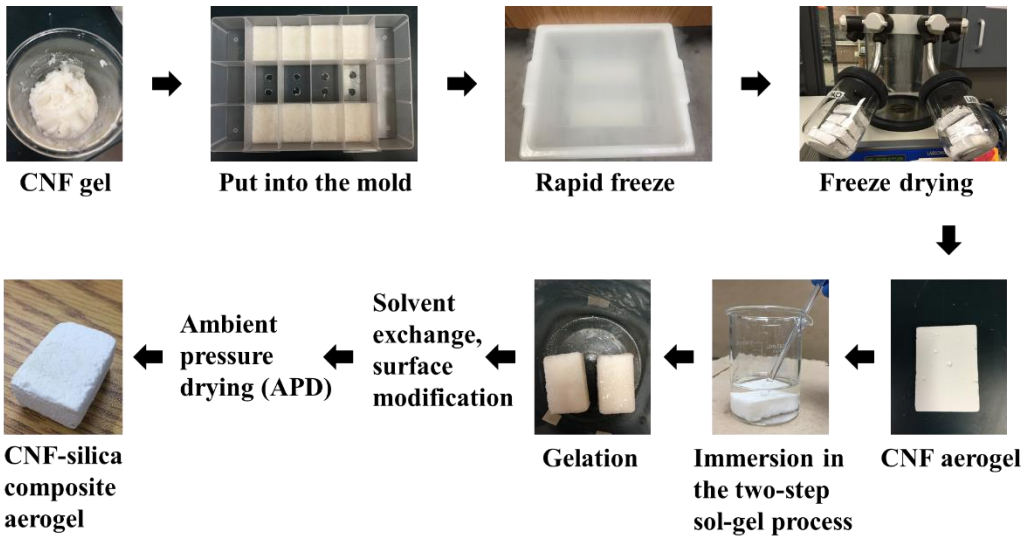

Figure S1. Preparation process of CNF-silica composite aerogel

Table S1. The BBD design matrix

Run	The concentration of CNF (wt%)	The concentration of TEOS (mol/L)	The pH value of condensation	Immersion time (min)
1	5	3.5	8	10
2	5	3.5	10	5
3	5	2.5	12	15
4	5	2.5	8	15
5	5	1.5	12	10
6	7	1.5	10	10
7	5	3.5	12	10
8	7	2.5	10	15
9	3	2.5	12	10
10	3	2.5	10	15
11	3	1.5	10	10
12	5	2.5	12	5
13	5	2.5	10	10
14	5	1.5	10	5
15	7	2.5	12	10
16	3	3.5	10	10
17	5	2.5	10	10
18	5	2.5	8	5
19	7	3.5	10	10
20	5	1.5	10	15
21	7	2.5	10	5
22	7	2.5	8	10
23	3	2.5	8	10
24	5	3.5	10	15
25	5	2.5	10	10
26	5	2.5	10	10
27	5	2.5	10	10
28	3	2.5	10	5
29	5	1.5	8	10

Table S2. ANOVA results of the quadratic model of silica content in CNF-silica aerogel

Source	SS	DF	MS	F-value	p-value
Model	16224.6967	14	1158.906907	16.07922525	< 0.0001
A	563.6905123	1	563.6905123	7.820910082	0.0143
B	124.9189916	1	124.9189916	1.733185462	0.2092
C	12926.25846	1	12926.25846	179.3450536	< 0.0001
D	612.8962435	1	612.8962435	8.503613784	0.0113
AB	31.15170479	1	31.15170479	0.432213558	0.5216
AC	53.44648131	1	53.44648131	0.741541884	0.4037
AD	819.8031533	1	819.8031533	11.37433859	0.0046
BC	229.8763412	1	229.8763412	3.189413615	0.0958
BD	104.8015911	1	104.8015911	1.454067085	0.2479
CD	124.7060832	1	124.7060832	1.730231469	0.2095
A^2	27.29502027	1	27.29502027	0.378704084	0.5482
B^2	227.4851307	1	227.4851307	3.156236824	0.0974
C^2	98.53565503	1	98.53565503	1.367130511	0.2618
D^2	181.7615642	1	181.7615642	2.52184633	0.1346
Residual	1009.05	14	72.07		
Lack of fit	996.62	10	99.66	32.08	0.0022
Pure error	12.43	4	3.11		

Table S3. ANOVA results of the quadratic model of density

Source	SS	DF	MS	F-value	p-value
Model	0.13	14	9.422×10^{-3}	9.32	< 0.0001
A	6.716×10^{-3}	1	6.716×10^{-3}	6.64	0.0219
B	6.480×10^{-5}	1	6.480×10^{-5}	0.064	0.8038
C	0.091	1	0.091	89.60	< 0.0001
D	4.794×10^{-3}	1	4.794×10^{-3}	4.74	0.0470
AB	3.057×10^{-4}	1	3.057×10^{-4}	0.30	0.5910
AC	6.875×10^{-5}	1	6.875×10^{-5}	0.068	0.7981
AD	6.700×10^{-3}	1	6.700×10^{-3}	6.63	0.0220
BC	6.137×10^{-3}	1	6.137×10^{-3}	6.07	0.0273
BD	5.774×10^{-4}	1	5.774×10^{-4}	0.57	0.4623
CD	5.556×10^{-4}	1	5.556×10^{-4}	0.55	0.4708
A^2	8.022×10^{-6}	1	8.022×10^{-6}	7.935×10^{-3}	0.9303
B^2	8.279×10^{-4}	1	8.279×10^{-4}	0.82	0.3808
C^2	0.013	1	0.013	13.18	0.0027
D^2	2.957×10^{-3}	1	2.957×10^{-3}	2.92	0.1093
Residual	0.014	14	1.011×10^{-3}		
Lack of fit	0.014	10	1.414×10^{-3}	436.83	< 0.0001
Pure error	1.295×10^{-5}	4	3.237×10^{-6}		

Table S4. ANOVA results of the quadratic model of compressive modulus

Source	SS	DF	MS	F-value	p-value
Model	160.51	14	11.46	1.18	0.3785
A	39.27	1	39.27	4.05	0.0637
B	3.06	1	3.06	0.32	0.5830
C	0.029	1	0.029	2.983×10^{-3}	0.9572
D	16.14	1	16.14	1.67	0.2177
AB	8.39	1	8.39	0.87	0.3678
AC	5.78	1	5.78	0.60	0.4528
AD	4.66	1	4.66	0.48	0.4995
BC	45.13	1	45.13	4.66	0.0488
BD	13.74	1	13.74	1.42	0.2534
CD	0.17	1	0.17	0.018	0.8961
A^2	8.58	1	8.58	0.89	0.3626
B^2	2.08	1	2.08	0.21	0.6506
C^2	6.41	1	6.41	0.66	0.4297
D^2	17.27	1	17.27	1.78	0.2031
Residual	135.62	14	9.69		
Lack of fit	133.52	10	13.35	25.37	0.0035
Pure error	2.10	4	0.53		

Table S5. ANOVA results of the quadratic model of compressive strength

Source	SS	DF	MS	F-value	p-value
Model	8.13	14	0.58	1.40	0.2685
A	1.91	1	1.91	4.60	0.0501
B	0.15	1	0.15	0.36	0.5586
C	0.23	1	0.23	0.55	0.4706
D	0.62	1	0.62	1.50	0.2404
AB	0.30	1	0.30	0.72	0.4113
AC	0.53	1	0.53	1.29	0.2758
AD	0.22	1	0.22	0.53	0.4801
BC	1.56	1	1.56	3.77	0.0727
BD	0.84	1	0.84	2.03	0.1766
CD	8.194×10^{-3}	1	8.194×10^{-3}	0.020	0.8902
A^2	0.46	1	0.46	1.11	0.3107
B^2	0.25	1	0.25	0.60	0.4497
C^2	0.68	1	0.68	1.64	0.2207
D^2	1.20	1	1.20	2.89	0.1111
Residual	5.80	14	0.41		
Lack of fit	5.27	10	0.53	3.93	0.0997
Pure error	0.54	4	0.13		

Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:CNF concen	is in range	4	6	1	1	3
B:TEOS conce	is in range	1.5	3.5	1	1	3
C:pH value	is in range	8	10	1	1	3
D:immersion tir	is in range	5	10	1	1	3
Silica content	is in range	40	50	1	1	3

Solutions

Number	CNF concentr	TEOS concent	pH value	immersion ti	Silica content	Desirability	
1	5.00	2.50	10.00	10.00	44.9559	1.000	Selected
2	4.88	1.62	9.05	5.46	40.0247	1.000	
3	4.43	1.55	9.26	9.42	45.5649	1.000	
4	5.00	3.50	10.00	5.00	40.6797	1.000	
5	5.15	1.65	9.85	5.38	49.8565	1.000	

Figure S2. Optimal data of the preparation condition using Design expert software

Characterization

Thermal conductivity

The thermal conductivity test was performed using a Conductometer (D300FX-D15), Tianjin Foreda Science & Technology Co., Ltd (Tianjin, China), following a double-plate method. The temperatures of the hot and cold plates were set at 35˚C and 15˚C, respectively.

Contact angel

The contact angles of the samples were determined from the water drop shape placed on the sample surface (JC2000D1 contact angel meter, POWERACH, Shanghai, China).