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Supplementary material

SUPPLEMENTARY MATERIAL TO

Development of a glass-ceramic composite reinforced with β-wollastonite synthesized via a hydrothermal method

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Table S-I. The most intense peaks in XRD spectra of wollastonite.

	Position / °2θ	*d*-spacing / Å	FWHM / °2θ	Backgr. / cts	Area / cts*°2θ	Crystallite size / Å	Micro strain / %
As-synthesized sample of wollastonite	22.80	3.89	0.2047	580.15	31.56	470	0.415
	25.36	3.51	0.1791	541.62	96.99	549	0.320
	26.91	3.31	0.2303	515.87	302.02	416	0.398
	28.92	3.08	0.1791	479.26	106.85	553	0.279
	30.17	2.96	0.1535	454.61	445.23	664	0.223
	36.48	2.46	0.2047	325.62	41.29	485	0.254
	39.18	2.29	0.156	295.71	201.59	706	0.163
	41.83	2.15	0.3744	278.76	280.44	216	0.500
	49.63	1.83	0.156	229.65	93.21	737	0.124
	53.29	1.71	0.3744	207.17	141.19	226	0.380
	Position / °2θ	*d*-spacing / Å	FWHM / °2θ	Backgr. / cts	Area / cts*°2θ	Crystallite size / Å	Micro strain / %
Calcined sample of wollasniteto	23.19	3.83	0.1535	610.89	114.8	655	0.292
	25.34	3.51	0.1535	560.4	256.45	657	0.267
	26.90	3.31	0.1791	527.31	414.11	550	0.301
	28.88	3.09	0.2047	485.04	238.59	475	0.325
	30.03	2.97	0.1535	460.53	501.07	663	0.224
	36.25	2.47	0.2047	351.64	117.99	484	0.256
	39.10	2.30	0.1791	313.28	138.35	567	0.203
	41.28	2.18	0.1279	283.91	78.91	861	0.127
	49.79	1.83	0.1535	231.04	58.97	707	0.129
	53.08	1.72	0.4605	208.03	195.23	219	0.393

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Table S-II. Textural parameters of as-synthesized wollastonite.

Sample Mass:	0.1844 g	Warm Free Space:	20.24 cm^3 Measured
Cold Free Space:	60.1 cm^3	Equilibration Interval:	10 s
Low Pressure Dose:	None	Sample Density:	1.0 gcm^{-3}
Automatic Degas:	No	/	/
Surface Area			m^2g^{-1}
Single point surface area			25
BET Surface Area			26
BJH Adsorption cumulative surface area			30
BJH Desorption cumulative surface area			30
Pore Volume			cm^3g^{-1}
Single point adsorption total pore volume			0.006
BJH Adsorption cumulative volume of pores			0.046
BJH Desorption cumulative volume			0.047
Pore Size			nm
Adsorption average pore diameter (4V/A by BET):			0.9
BJH Adsorption average pore diameter (4V/A):			6.1
BJH Desorption average pore diameter (4V/A):			6.3

Table S-III. Mode assignments of the Raman-active vibrational modes of the wollastonite.

Assignment	As-prepared (cm^{-1})	Calcined (900 °C) (cm^{-1})
Asymmetric stretching of Si-O	992	971
Symmetric stretching of Si-O	/	894
Symmetric stretching of Si-O	/	860
Symmetric bending vibration of Si-O-Si	648	636
Bending vibrations of O-Si-O	408	404

SEM ANALYSES OF THE VARIOUS SAMPLES

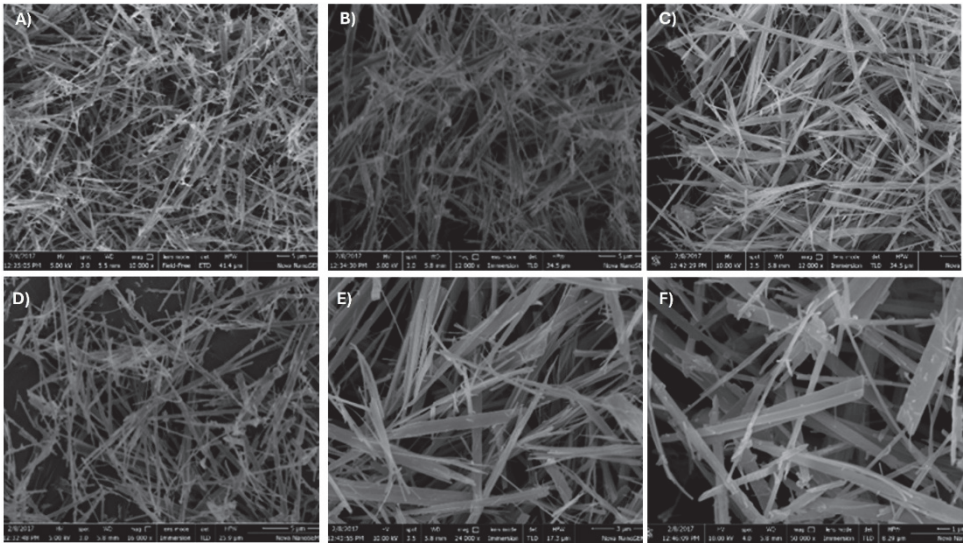


Fig. S-1. SEM micrographs of as-prepared wollastonite at different magnifications: A) 10000 x; B) 12000 x; C) 12000 x; D) 16000 x; E) 24000 x; F) 50000 x.

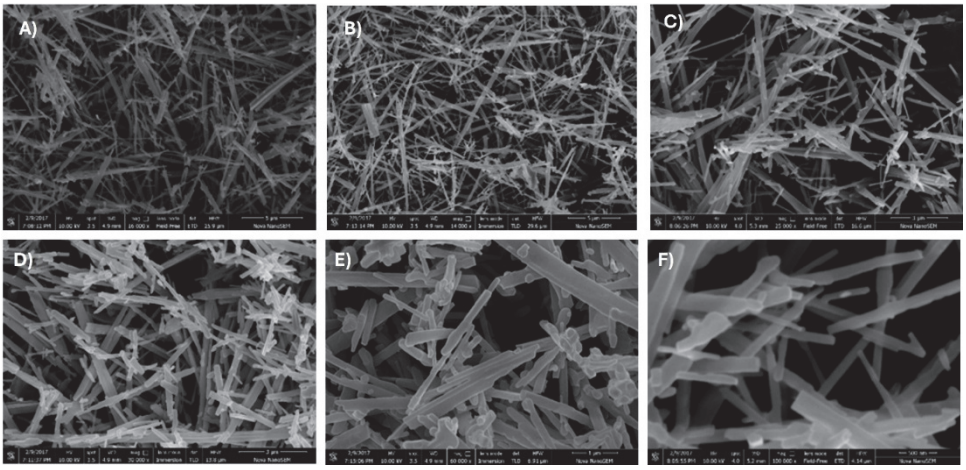


Fig. S-2. SEM micrographs of calcined wollastonite at different magnifications: A) 10000 x; B) 14000 x; C) 25000 x; D) 30000 x; E) 60000 x; F) 100000 x.

BG 1000°C 15

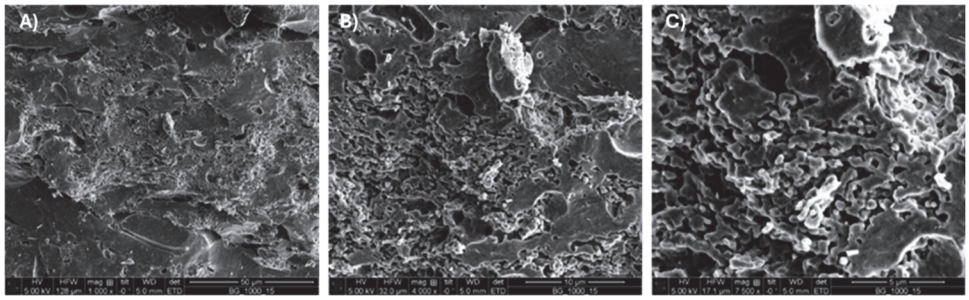


Figure S-3. SEM micrographs of the sintered bioglass treated at 1000 °C for 15 minutes (BG 1000°C 15) at different magnifications: A) 1000 x; B) 4000 x; C) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

Porosity measurements were conducted at -195.8°C after degassing samples at 250°C under vacuum to remove adsorbates. Data analysis was performed using "ASiQwin" by Quantachrome Instruments.

EDX analysis:

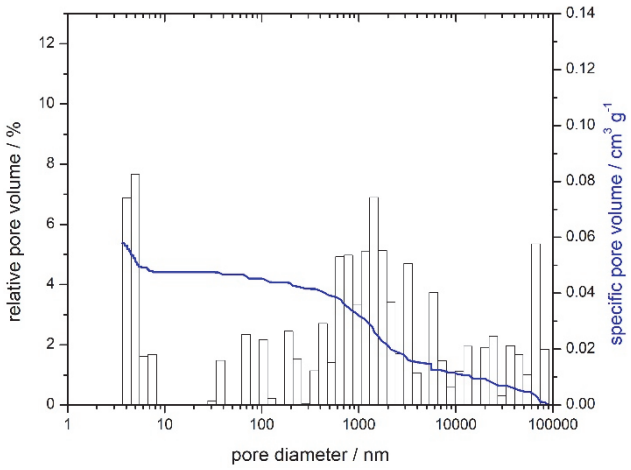
O: 39.4 wt.-% Si: 21.8 wt.-%
Na: 12.3 wt.-% P: 1.3 wt.-%
Ca: 25.2 wt.-%

XRD analysis:

Combeite: 96 wt.-%
Disodium calcium silicate: 4 wt.-%
Rwp: 9

Texture data:

BET: <1 m^2g^{-1}
Vp: 0,058 cm^3g^{-1}
dp: 9671 nm (average diameter)
Porosity: 11.3



BG 1000°C 30

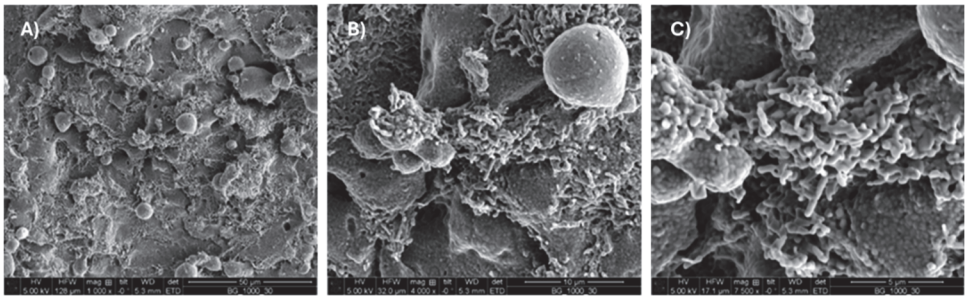


Figure S-4. SEM micrographs of the sintered bioglass treated at 1000 °C for 30 minutes (BG 1000°C 30) at different magnifications: A) 1000 x; B) 4000 x; C) 7500 x.

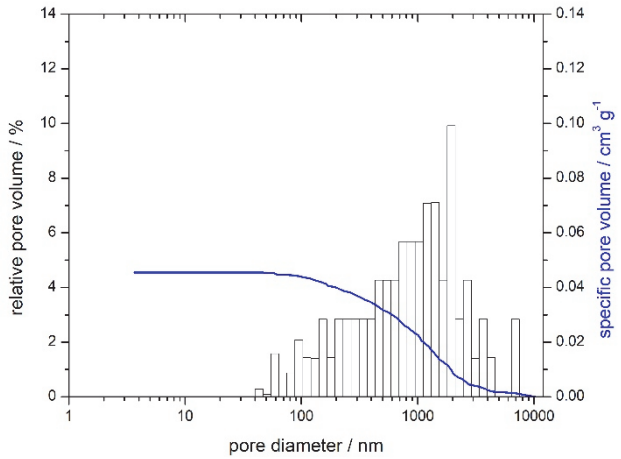
Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:

O:	40.4 wt.-%	Si:	21.0 wt.-%
Na:	15.2 wt.-%	P:	2.0 wt.-%
Ca:	21.5 wt.-%		

XRD analysis:
Combeite: 99.5 wt.-%
Disodium calcium silicate: <1 wt.-%
Rwp: 11

Texture data:
BET: <1 m^2g^{-1}
Vp: 0.036 cm^3g^{-1}
dp: 1348 nm (average diameter)
Porosity: 7.4 %



BG 1000°C 55

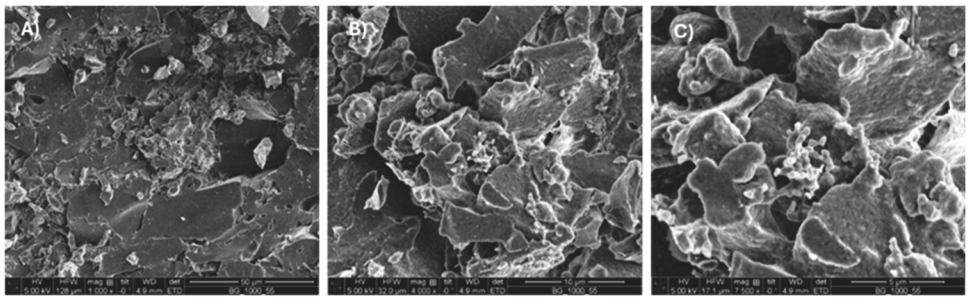


Figure S-5. SEM micrographs of the sintered bioglass treated at 1000 °C for 55 minutes (BG 1000°C 55) at different magnifications: A) 1000 x; B) 4000 x; C) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:

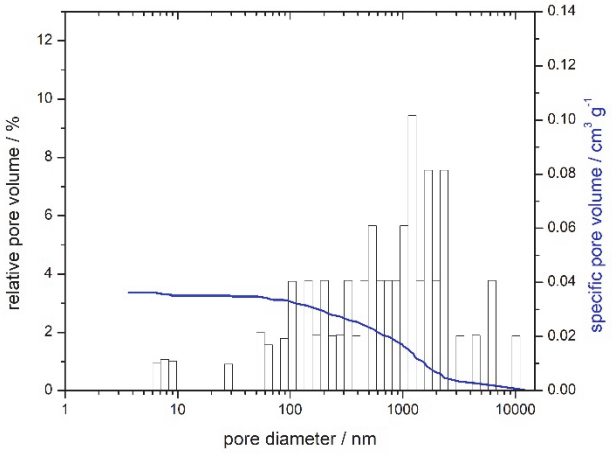
O:	40.4 wt.-%	Si:	21.0 wt.-%
Na:	15.2 wt.-%	P:	2.0 wt.-%
Ca:	21.5 wt.-%		

XRD analysis:

Combeite:	99.5 wt.-%
Disodium calcium silicate:	<1 wt.-%
Rwp:	11

Texture data:

BET:	<1 m^2g^{-1}
Vp:	0.036 cm^3g^{-1}
dp:	1348 nm (average diameter)
Porosity:	7.4 %



BG 1000°C 85

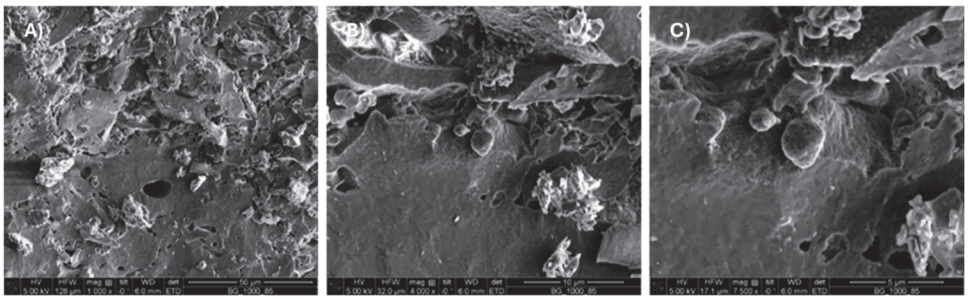


Figure S-6. SEM micrographs of the sintered bioglass treated at 1000 °C for 85 minutes (BG 1000°C 85) at different magnifications: A) 1000 x; B) 4000 x; C) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:

O:	44.5 wt.-%	Si:	20.5 wt.-%
Na:	17.4 wt.-%	P:	1.7 wt.-%
Ca:	15.9 wt.-%		

XRD analysis:

Combeite: 100 wt.-%

Disodium calcium silicate: 0 wt.-%

Rwp:11

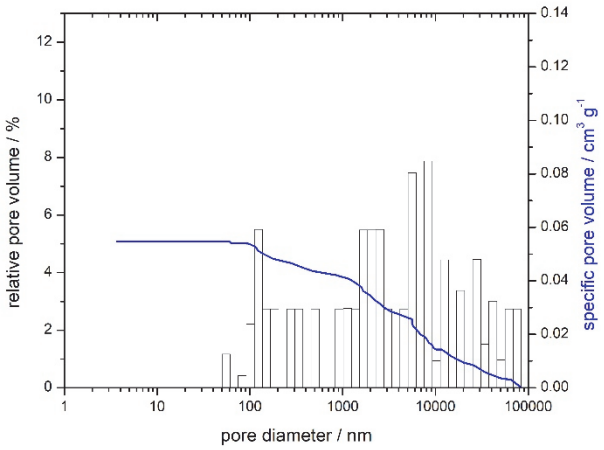
Texture data:

BET: <1 m^2g^{-1}

Vp: 0.055 cm^3g^{-1}

dp: 11104 nm (average diameter)

Porosity: 10.7 %



COM 1000°C 15

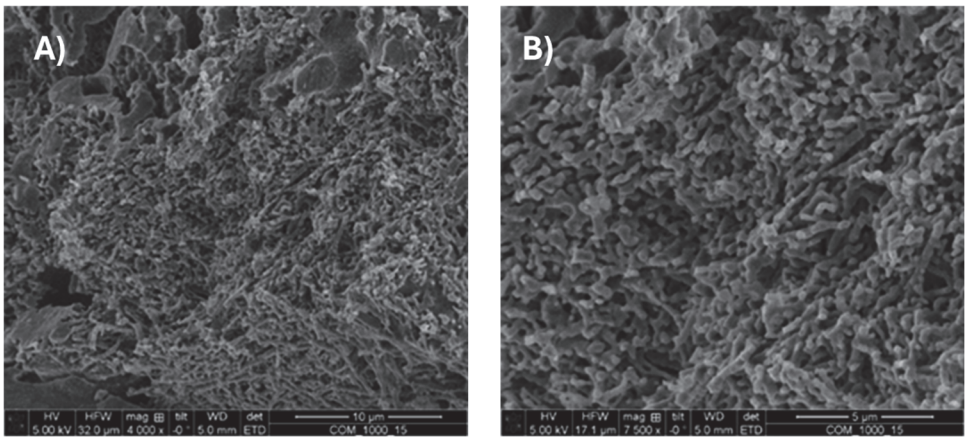


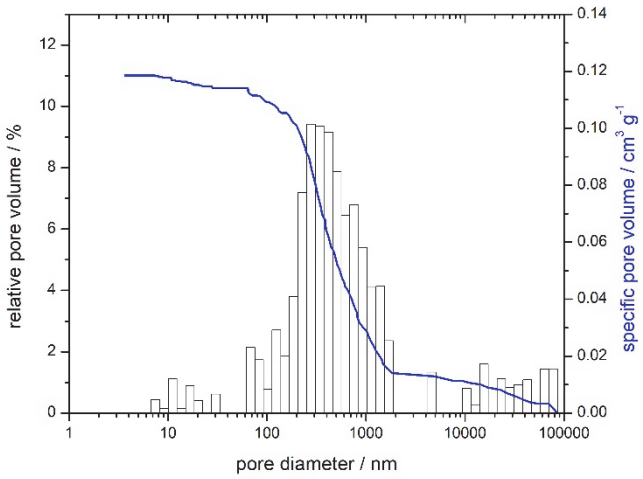
Figure S-7. SEM micrographs of the composite treated at 1000 °C for 15 minutes (COM 15) at different magnifications: A) 4000 x; B) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:
O: 40.0 wt.-% Si: 21.8 wt.-%
Na: 12.5 wt.-% P: 1.4 wt.-%
Ca: 24.3 wt.-%

XRD analysis:
Combeite: 98.5 wt.-%
Disodium calcium silicate: 1.5 wt.-%
Rwp: 10

Texture data:
BET: <1 m^2g^{-1}
Vp: 0.119 cm^3g^{-1}
dp: 4256 nm (average diameter)
Porosity: 20.7 %



COM 1000°C 30

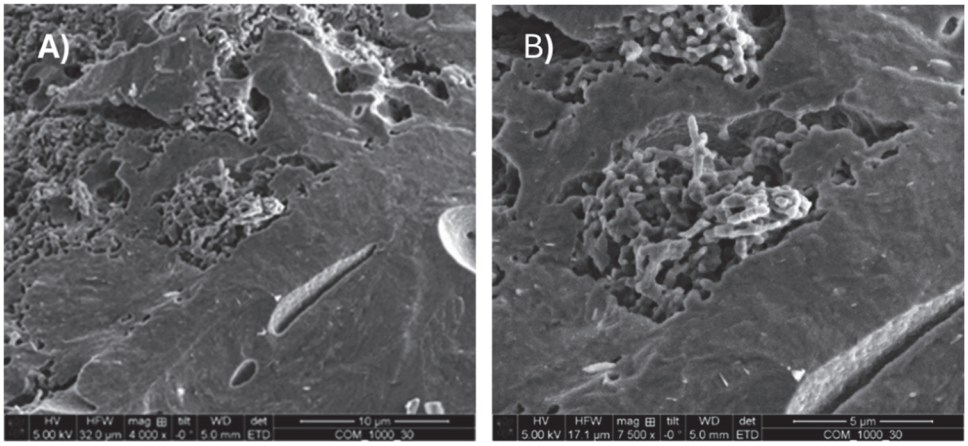


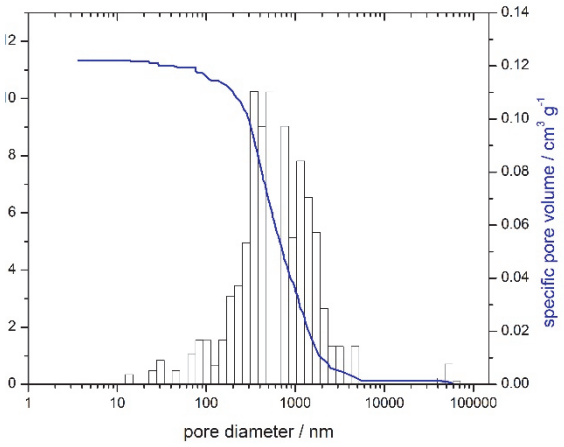
Figure S-8. SEM micrographs of the composite treated at 1000 °C for 30 minutes (COM 30) at different magnifications: A) 4000 x; B) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:
O: 45.2 wt.-% Si: 21.1 wt.-%
Na: 14.5 wt.-% P: 1.3 wt.-%
Ca: 17.8 wt.-%

XRD analysis:
Combeite: 98.5 wt.-%
Disodium calcium silicate: 1.5 wt.-%
Rwp:

Texture data:
BET: <1 m^2g^{-1}
Vp: 0.122 cm^3g^{-1}
dp: 1368 nm (average diameter)
Porosity: 21.2 %



COM 1000°C 55

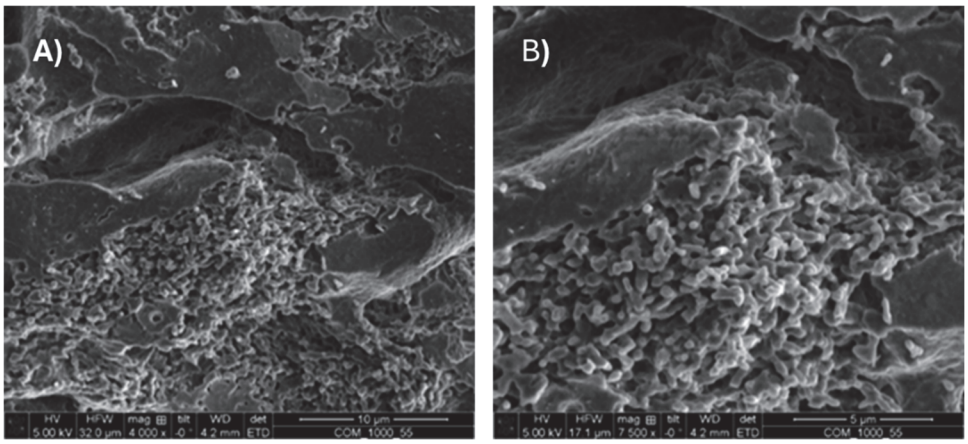


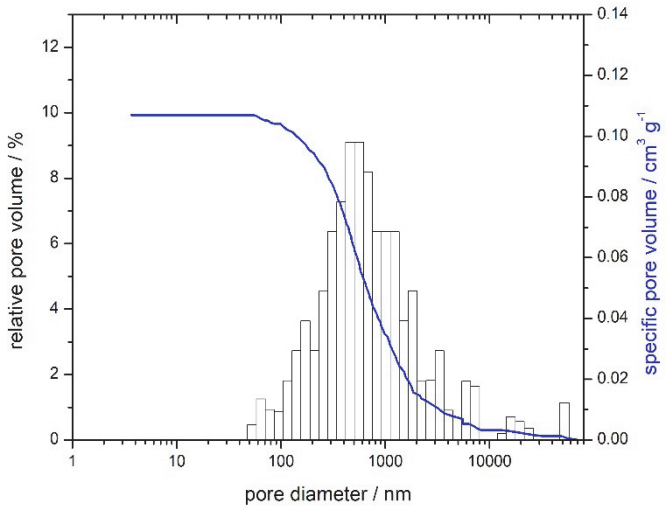
Figure S-9. SEM micrographs of the composite treated at 1000 °C for 55 minutes (COM 55) at different magnifications: A) 4000 x; B) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:
O: 40.0 wt.-% Si: 21.3 wt.-%
Na: 12.5 wt.-% P: 1.2 wt.-%
Ca: 25.1 wt.-%

XRD analysis:
Combeite: 99.5 wt.-%
Disodium calcium silicate: <1 wt.-%
Rwp: 11

Texture data:
BET: <1 m^2g^{-1}
Vp: 0.107 cm^3g^{-1}
dp: 1956 nm (average diameter)
Porosity: 19 %



COM 1000°C 85

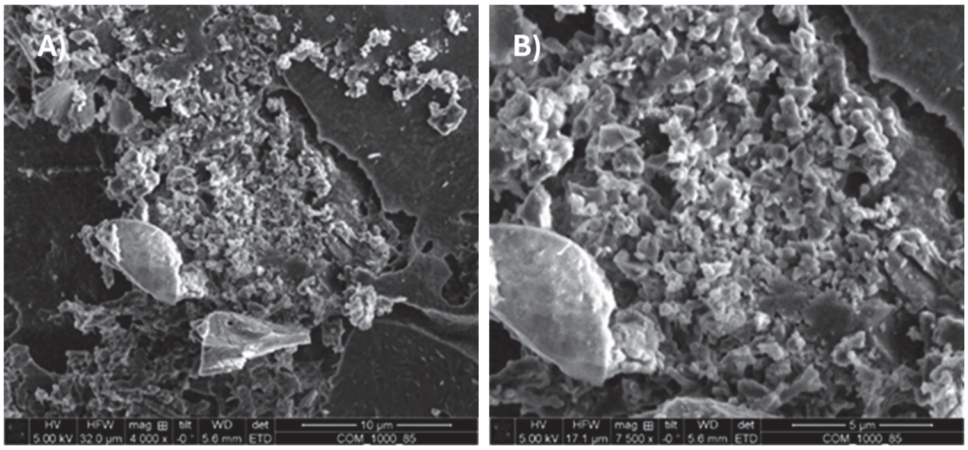


Figure S-10. SEM micrographs of the composite treated at 1000 °C for 85 minutes (COM 85) at different magnifications: A) 4000 x; B) 7500 x.

Structural and textural parameters obtained by EDX, XRD, and BET analysis are given below:

EDX analysis:

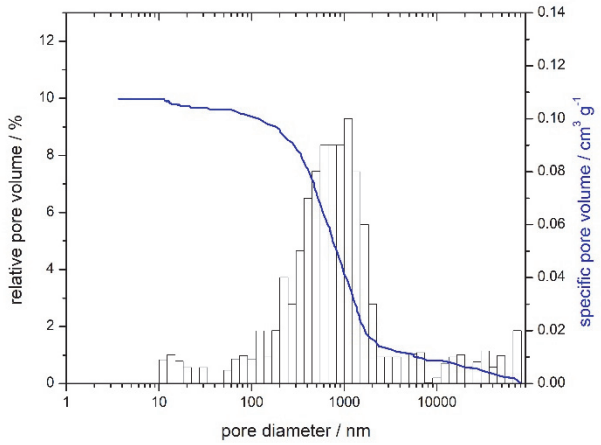
O: 45.1 wt.-% Si: 21.1 wt.-%
Na: 14.9 wt.-% P: 1.1 wt.-%
Ca: 17.8 wt.-%

XRD analysis:

Combeite: 99.5 wt.-%
Disodium calcium silicate: <1 wt.-%
Rwp: 11

Texture data:

BET: <1 m^2g^{-1}
Vp: 0.108 cm^3g^{-1}
dp: 3928 nm (average diameter)
Porosity: 19.1 %



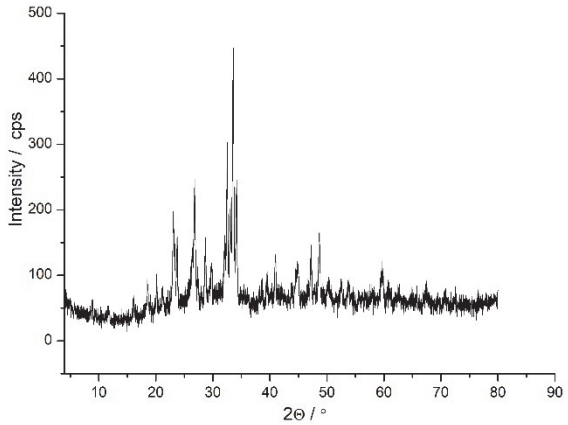


Figure S-11. XRD pattern of COM 30 after interaction with the SBF solution. Pekas at 26.0°, 31.8°, and 33.1 correspond to the (002), (211), and (300) crystal planes of hydroxyapatite.