

Fibre reinforced fly-ash based geopolymer composites

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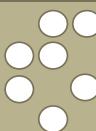
Engineering Ceramics Department

Jožef Stefan Institute, Ljubljana, Slovenia



Scope of the Study

- A main objective of the cement industry is a reduction of the clinker factor, as the high CO₂-emission of portland cement (CEM I) are directly linked to the clinker factor. The cements recently developed cause either strong embrittlement or reduction of the resistance to freeze thaw cycles when used in fibre cement production.
- To evaluate the possibility of use of various types of fibers for the reinforcement of geopolymmer matrix



Materials

- Fly ash
 - Local source (thermal electrical plant Trbovlje, Slovenia)
 - Composition:

Component	wt. %	Component	wt. %
SiO ₂	48,10	MgO	2,70
Al ₂ O ₃	25,00	SO ₃	1,60
Fe ₂ O ₃	8,80	Na ₂ O	2,00
CaO	7,10	K ₂ O	2,50

- milling (24 h in a ball mill), sieving 325 mesh
- NaOH (16 M solution, Alpha Aesar)
- Sodium silicate solution (NAVS 48, Silkem, Kidričevo)

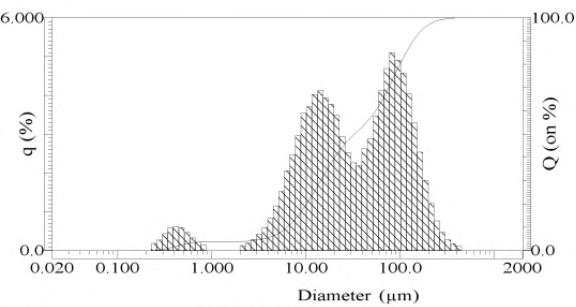


Materials

"Jožef Stefan" Institute, Ljubljana, Slovenija
Engineering Ceramics Department

Particle size distribution analysis

Filename	:elektrofiltrski pepel
ID#	:201010191502532
Circulation Speed	:12
Ultra sonic	:05:00 (7)
Laser T%	: 87.8(%)
Lamp T%	: 81.4(%)
Distribution Base	:Volume
R.R.Index	:115a0001
Sample Name	:elektrofiltrski pepel
Material	:
Dispersant	:dolapix

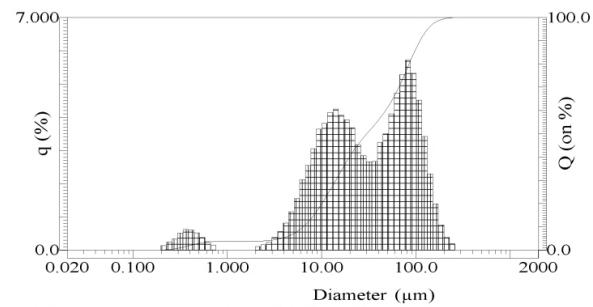


S.P. Area	: 8695.9(cm ² /cm ³)
Median	: 32.7356(μm)
Diameter on %	(1)5.000 (%) - 3.5037(μm) (2)10.00 (%) - 6.2787(μm) (3)20.00 (%) - 10.0977(μm) (4)30.00 (%) - 14.2759(μm) (5)40.00 (%) - 20.2565(μm) (6)60.00 (%) - 54.7037(μm) (7)70.00 (%) - 75.7463(μm) (8)80.00 (%) - 99.4465(μm) (9)90.00 (%) - 136.8208(μm) (10)95.00 (%) - 173.0932(μm)
Mean	: 56.9335(μm)
Variance	: 3595.0(μm ²)
S.D.	: 59.9581(μm)
Mode	: 83.0021(μm)
R Parameter	: 7.4658E-2

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Particle size distribution analysis

Filename	:elektrofiltrski pepel(po sejanju)
ID#	:201010221052538
Circulation Speed	:12
Ultra sonic	:05:00 (7)
Laser T%	: 96.0(%)
Lamp T%	: 87.4(%)
Distribution Base	:Volume
R.R.Index	:115a0001
Sample Name	:elektrofiltrski pepel-sejanje
Material	:
Dispersant	:dolapix

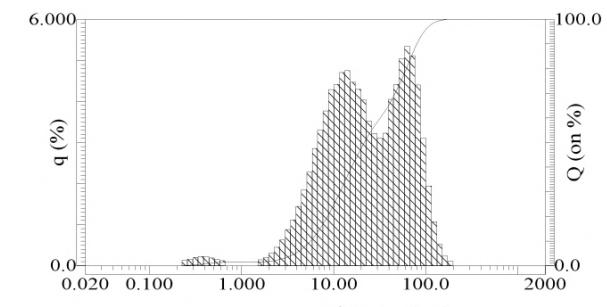


S.P. Area	: 9144.3(cm ² /cm ³)
Median	: 30.0795(μm)
Diameter on %	(1)5.000 (%) - 3.7288(μm) (2)10.00 (%) - 6.3760(μm) (3)20.00 (%) - 10.0784(μm) (4)30.00 (%) - 14.1275(μm) (5)40.00 (%) - 19.7628(μm) (6)60.00 (%) - 47.7251(μm) (7)70.00 (%) - 66.0611(μm) (8)80.00 (%) - 84.9634(μm) (9)90.00 (%) - 110.8129(μm) (10)95.00 (%) - 133.1550(μm)
Mean	: 47.4594(μm)
Variance	: 1962.7(μm ²)
S.D.	: 44.3021(μm)
Mode	: 82.8019(μm)
R Parameter	: 7.2096E-2

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Particle size distribution analysis

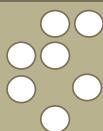
Filename	:elektrofiltrski pepel mlet 24h
ID#	:201203300922834
Circulation Speed	:8
Ultra sonic	:01:53 (7)
Laser T%	: 90.9(%)
Lamp T%	: 92.0(%)
Distribution Base	:Volume
R.R.Index	:115a0001
Sample Name	:elektr.pepel po mletju 24h
Material	:
Dispersant	:



S.P. Area	: 6556.2(cm ² /cm ³)
Median	: 21.6036(μm)
Diameter on %	(1)5.000 (%) - 3.8378(μm) (2)10.00 (%) - 5.6193(μm) (3)20.00 (%) - 8.6118(μm) (4)30.00 (%) - 11.7827(μm) (5)40.00 (%) - 15.7340(μm) (6)60.00 (%) - 32.1583(μm) (7)70.00 (%) - 46.7351(μm) (8)80.00 (%) - 61.7549(μm) (9)90.00 (%) - 80.5807(μm) (10)95.00 (%) - 96.5874(μm)
Mean	: 34.5784(μm)
Variance	: 989.00(μm ²)
S.D.	: 31.4484(μm)
Mode	: 63.1527(μm)
R Parameter	: 5.7731E-2

Materials

- Fibers (reinforcement)
 - 4 and 6 mm PVA fibers – PVA A-08 – 2,0 dtex, Kuraray Co. Ltd.
 - 4 mm PVA fibers – PVA A-20 Kuralon, Kuraray Co. Ltd.
 - 6 mm PVA fibers – PVA A-70 Kuralon, Kuraray Co. Ltd.
 - 4 mm PP fibers – E12C, Daiwabo
- Glass fibers
 - Cem-FIL, Owens Corning
 - Cem-FIL Anti-Crack HD, Owens Corning
- Carbon fibers, milled (~0,7 mm), pitch type, Kureha, Japan
- Carbon fibers 3 mm, 6 mm, pan type, Kureha, Japan
- Wolastonite, natural source
- Steel Fiber SO1, Green Steel, Poland

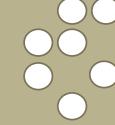
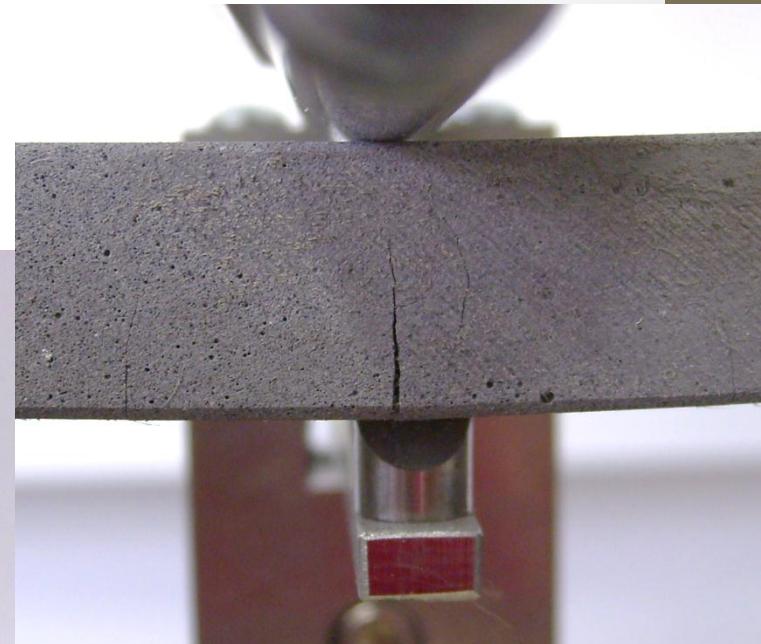
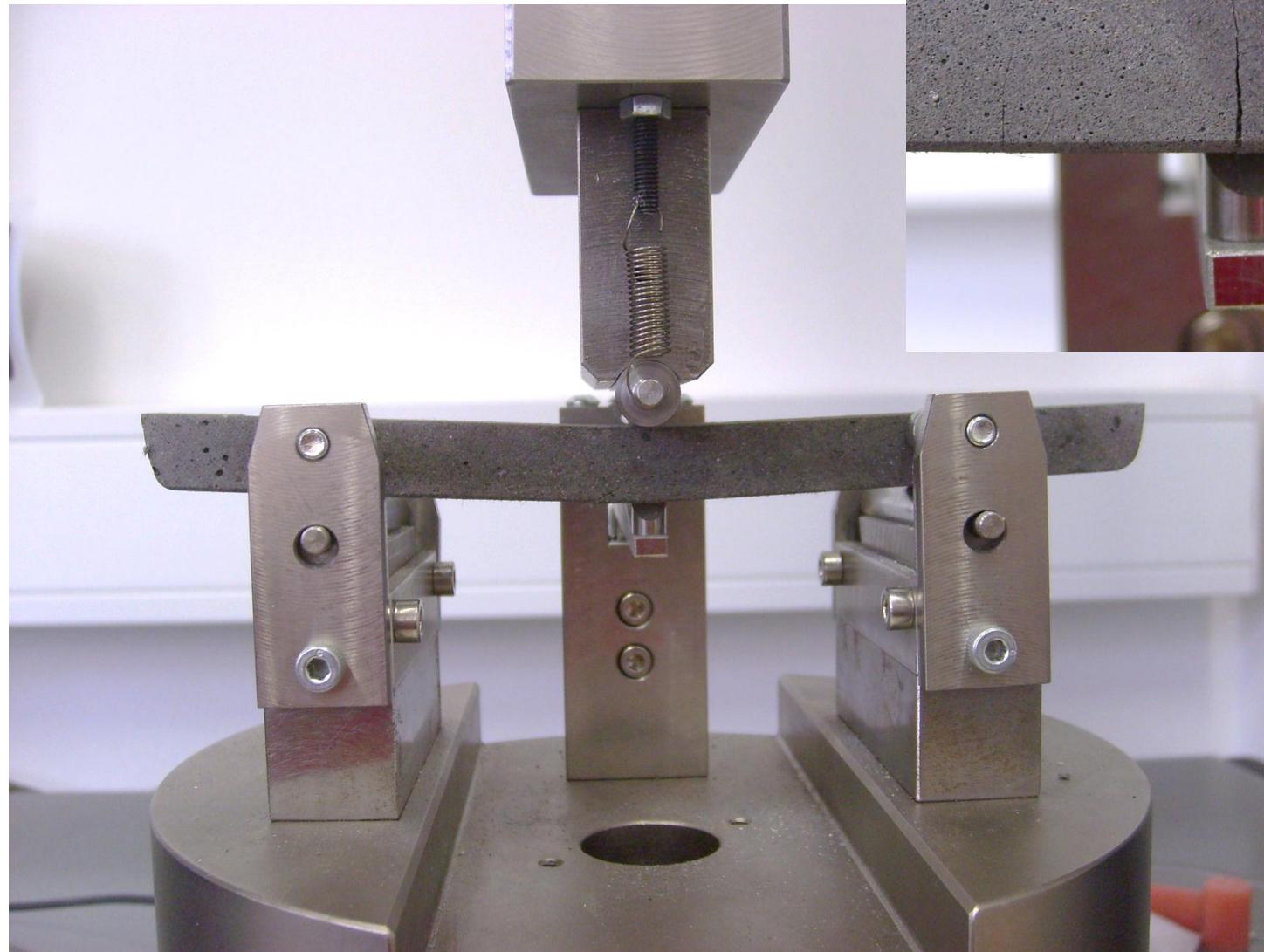


Processing and characterization

- The constituents were mixed
- Samples were de-aired by vibration for 10 min
- All of the samples were cured at RT for 24 hours, followed by 80 °C for 24 hours in closed molds
- Samples were left in the molds until testing
- Compressive strength (Quasar 50, Galdabini, Italy)
- 3-point bending strength (Quasar 50, Galdabini, Italy)
 - 100 mm span
 - Sample size approx. 12x12x120 mm³
- SEM characterization (SEM 5800, JEOL, Japan)



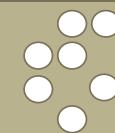
3-point bending



Matrix compositions

- Matrix materials
 - compressive strength after 7 days,
 - curing 24 h at RT + 24 hours at 80 °C

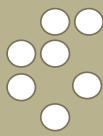
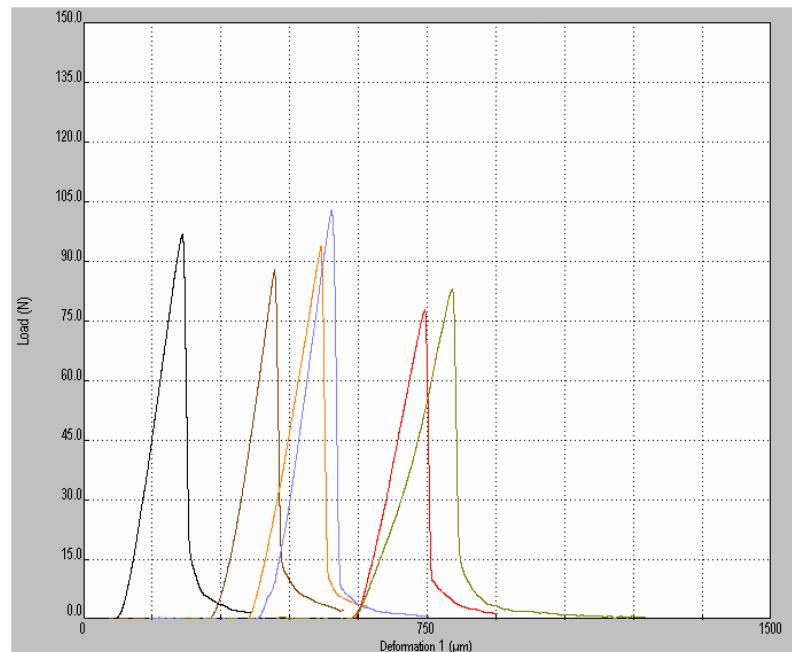
series	AA/FA	N a O H , conc. moldm ⁻³	Na ₂ O/Al ₂ O ₃	SiO ₂ /Al ₂ O ₃	Na ₂ O/SiO ₂	Compressive strength, MPa
LP1	0,3	8	0,47	3,46	0,08	6,75
	0,3	10	0,57	3,34	0,10	3,96
	0,3	12	0,68	3,22	0,13	4,54
LP2	0,35	8	0,53	3,54	0,1	26,62
	0,35	10	0,66	3,39	0,12	36,36
	0,35	12	0,80	3,25	0,15	5,88
LP3	0,4	8	0,61	3,62	0,10	40,93
	0,4	10	0,75	3,46	0,13	44,66
	0,4	12	0,89	3,30	0,16	8,33



Matrix - mechanical properties

- Matrix
 - Compressive strength after 7 days: 33 Mpa
 - 3 point bending strength (Mpa)

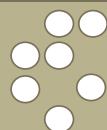
	7 days	28 days
EP-0	3,22	6,49



Composites - mechanical properties

- PVA fibers reinforced composites

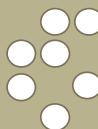
3 point bending strength (MPa)	7 days	28 days
EP+1%PVA-A8 4mm	5,12	6,22
EP+1,5%PVA-A8 4mm	5,05	9,14
EP+2%PVA -A8 4mm	4,48	3,07
EP+1%PVA-A20	7,28	10,56
EP+1%PVA-A70	8,95	8,40
EP+1,5%PVA-A70	7,81	9,73
EPsieved+1%PVA-A70	8,93	10,67
EPmilled+1%PVA-A70	4,93	8,89



Composites - mechanical properties

- PVA fibers reinforced composites – fiber combinations

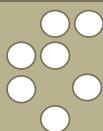
3 point bending strength (MPa)	7 days	28 days
EP+1%PVA+2%Wol	4,55	8,14
EP+1%PVA+1%Cf	7,17	7,61
EP+1%PVAA20+1%Cf	7,52	5,52
EP+1%PVA-A70+1%Cf	9,49	8,18
EP+1%PVA-A70+2%Wol	8,61	10,70



Composites - mechanical properties

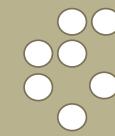
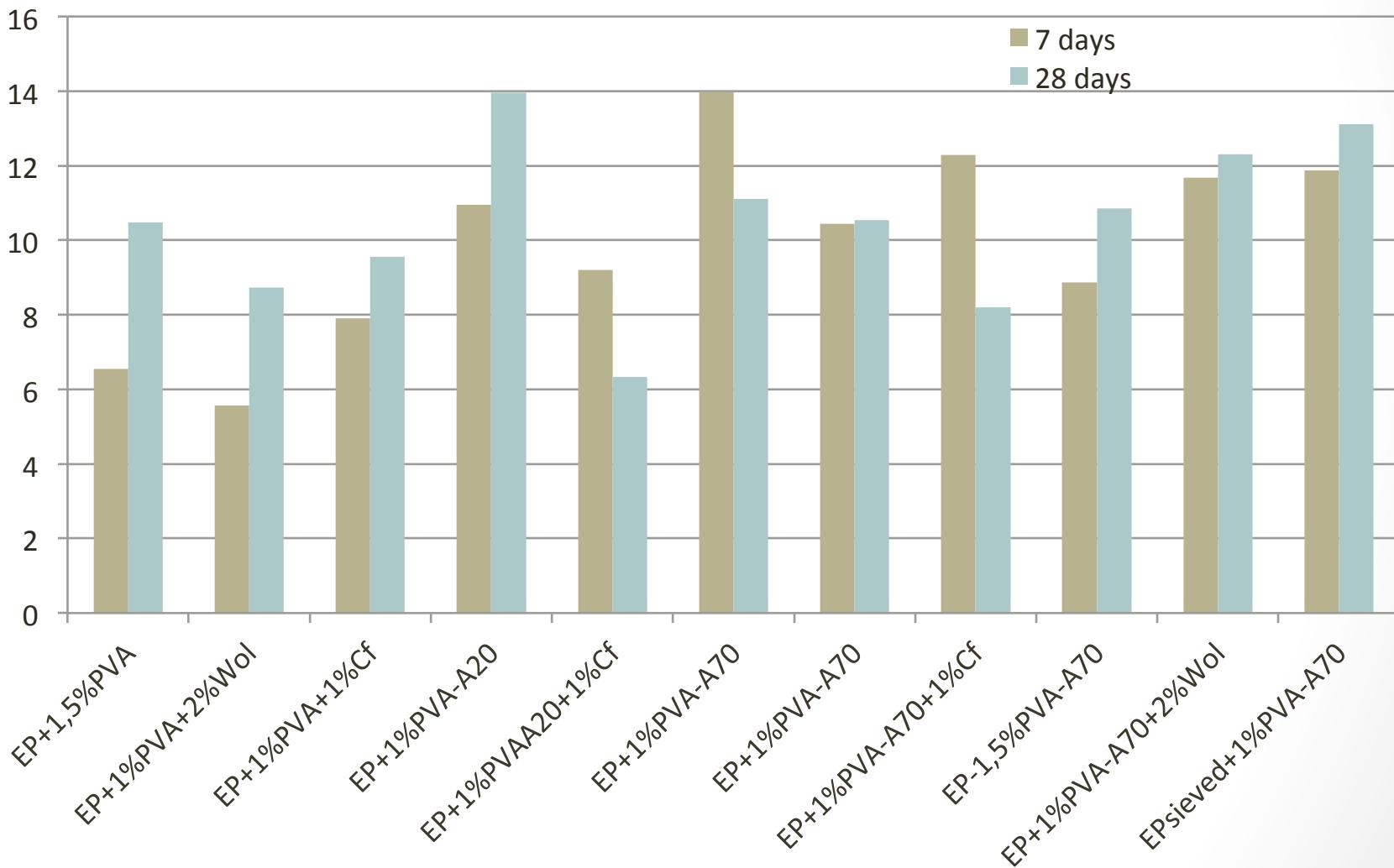
- other fibers reinforced composites

3 point bending strength (MPa)	7 days	28 days
EP+2%GF-Cem-FIL	5,08	4,49
Ep+4%GF-Cem-FIL	6,74	5,38
EP+2%GF-Cem-FIL Anticrack	5,67	6,79
EP+3,5% Steel Fiber SO1	5,11	-
EP+1% PP E12C	4,38	-



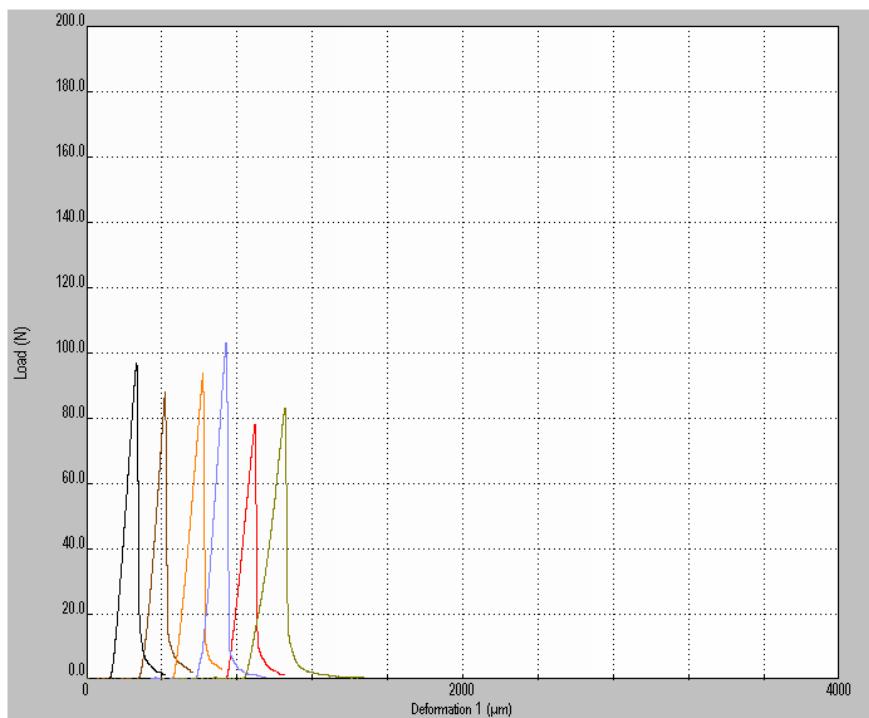
Composites - mechanical properties

- Maximum obtained values

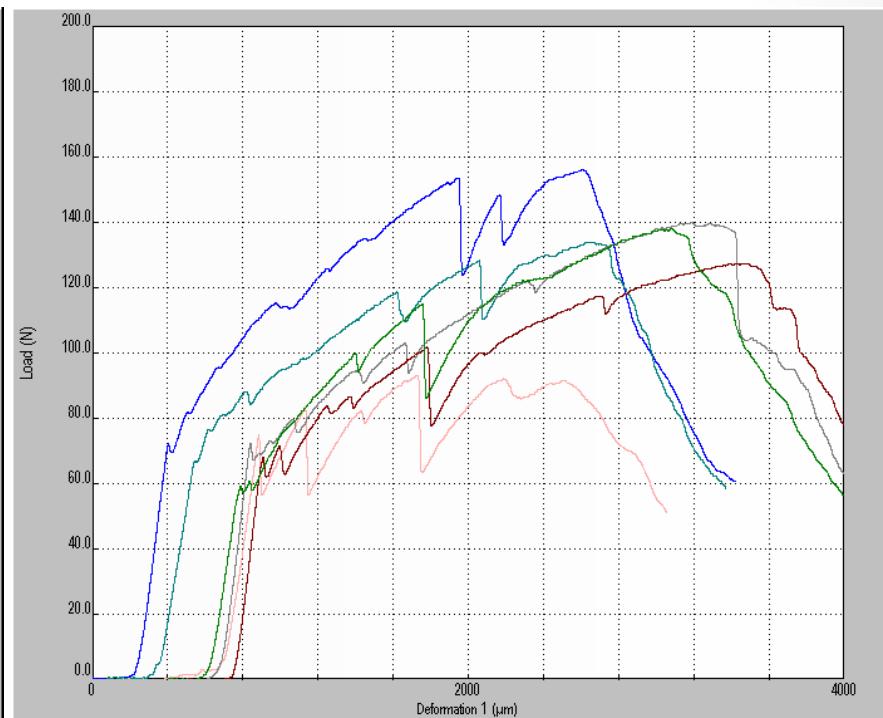


Composites - mechanical properties

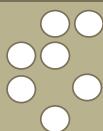
- Fracture curves



EP-0

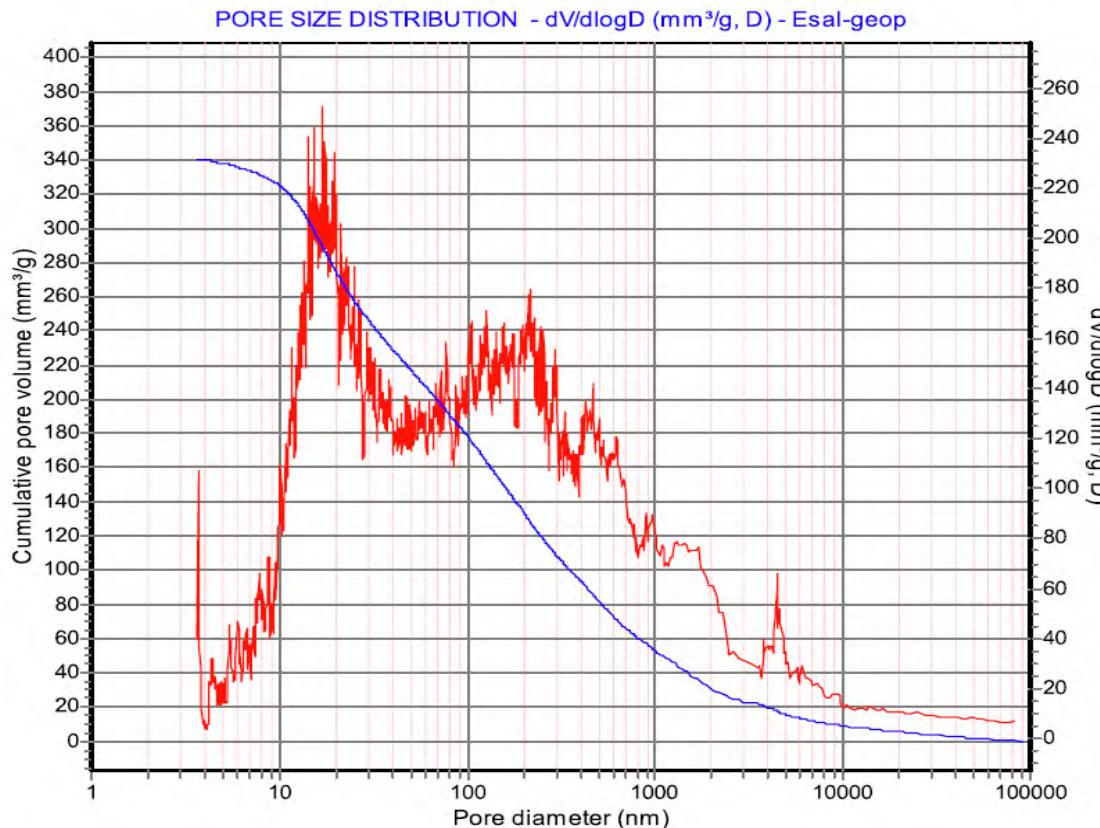


EP+1%PVA-A70



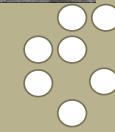
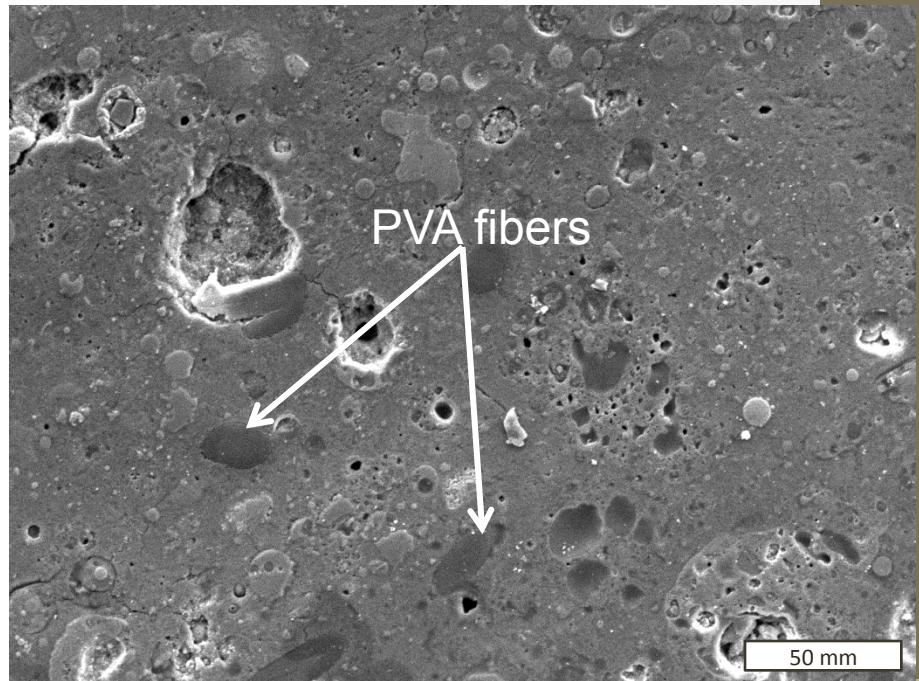
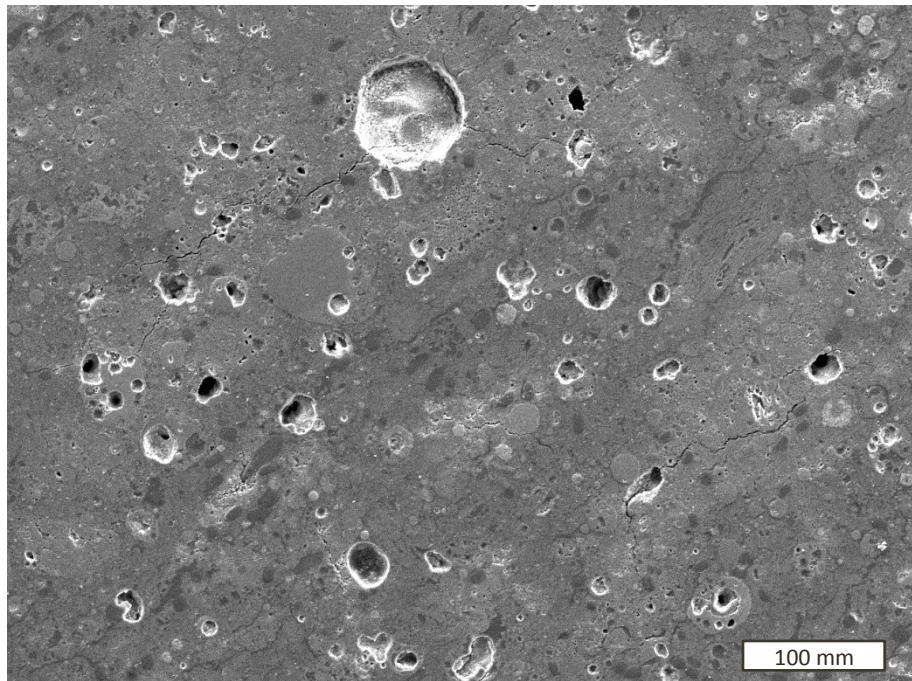
Composites – density & porosity

- Measurement of density and porosity
 - Sample: EP+1%PVA-A70
 - Apparent density: 2,3490 g/cm³



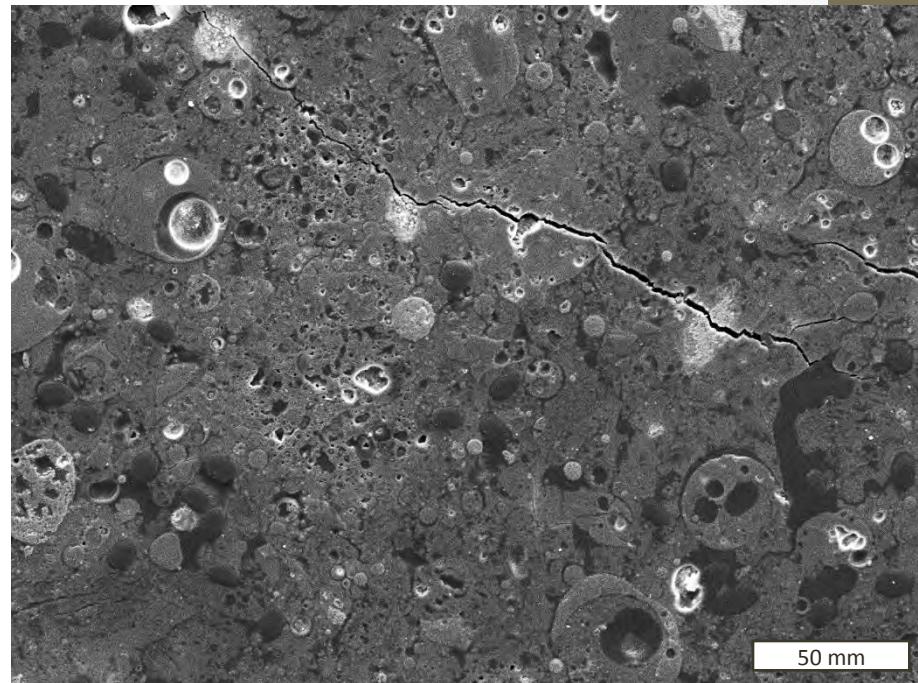
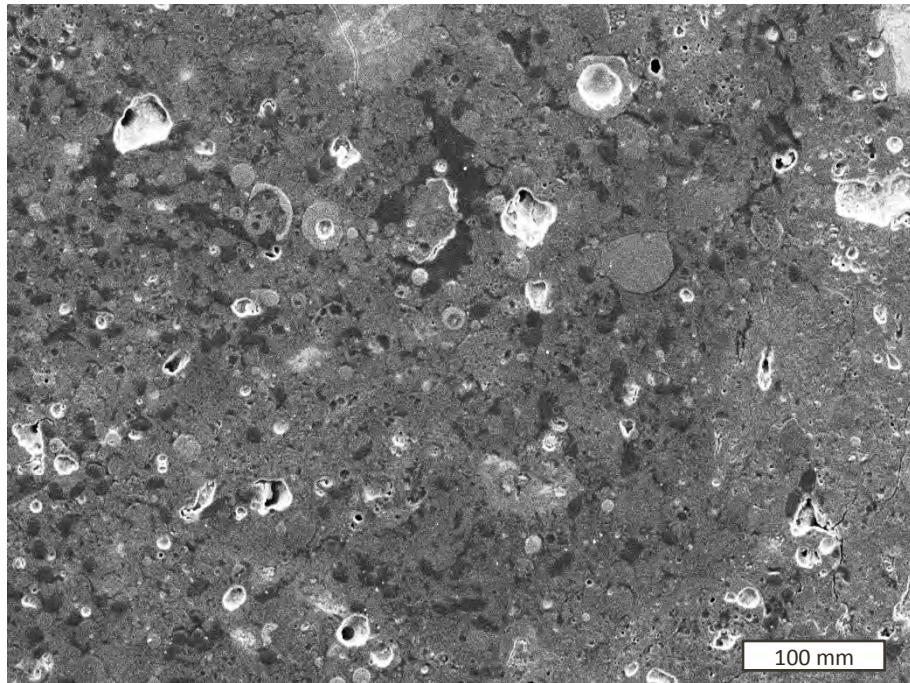
Microstructure

- 1 wt.% of PVA A8 4 mm fibers



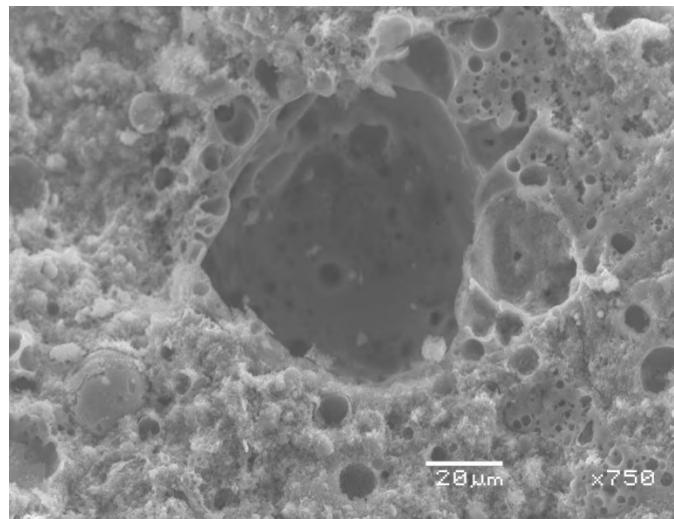
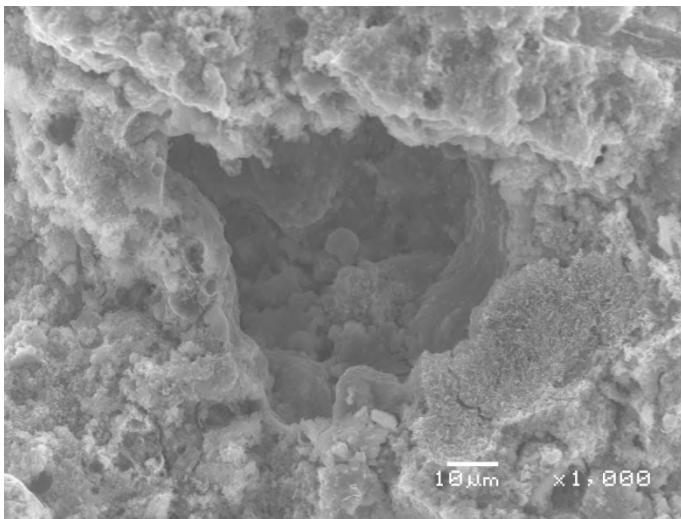
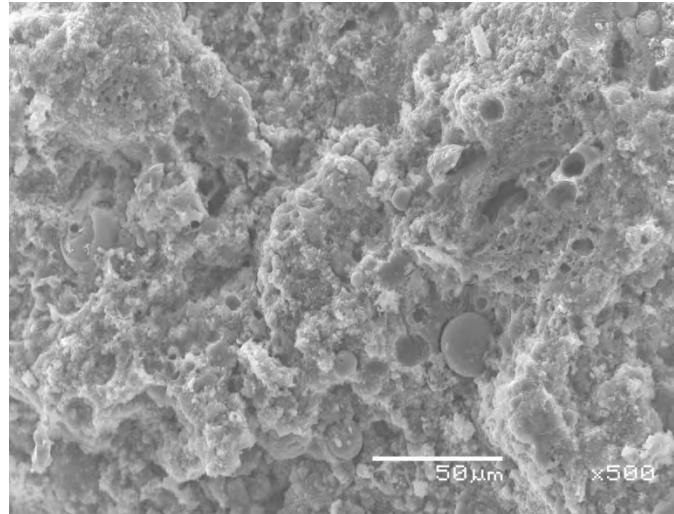
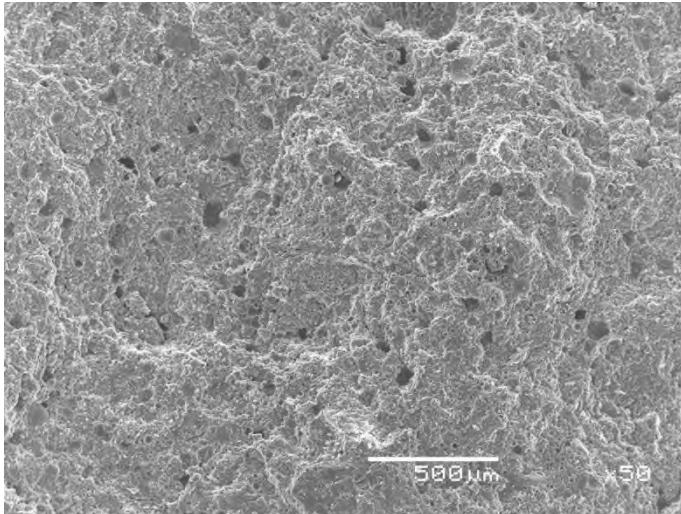
Microstructure

- 2 wt.% of PVA fibers



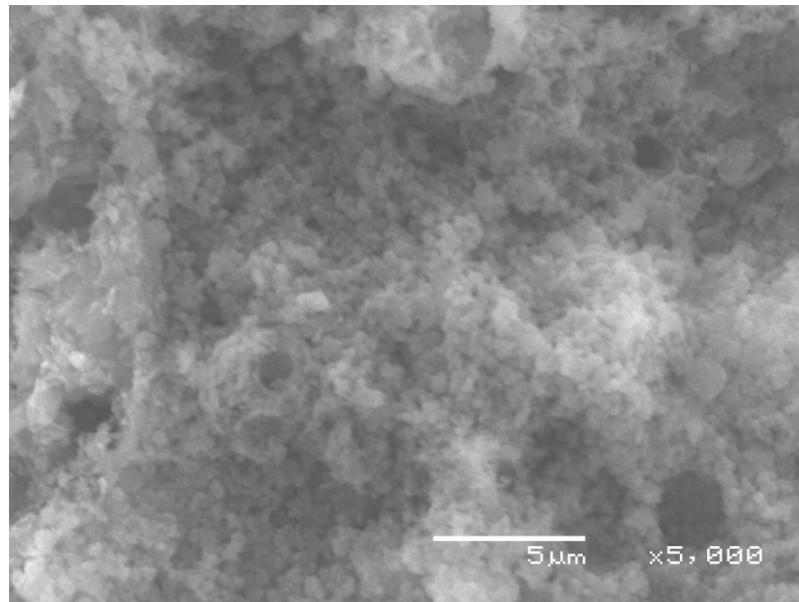
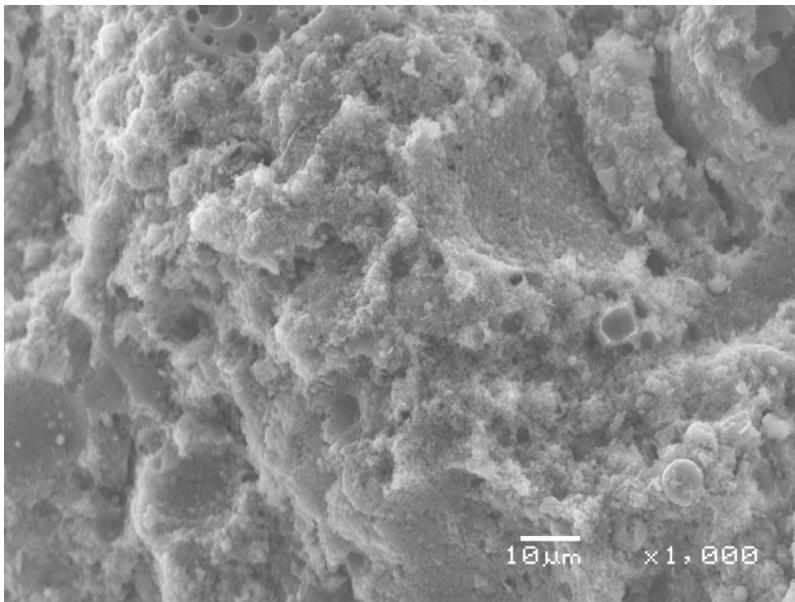
Fracture Surfaces

EP-0



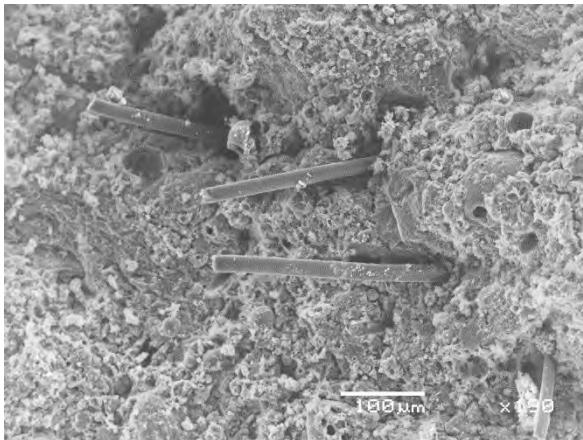
Fracture Surfaces

EP-0

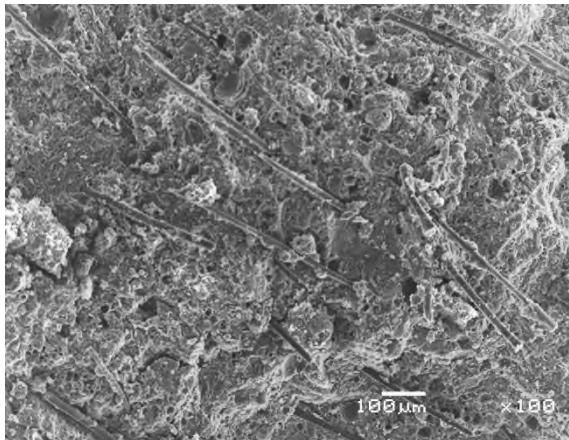


Fracture surfaces

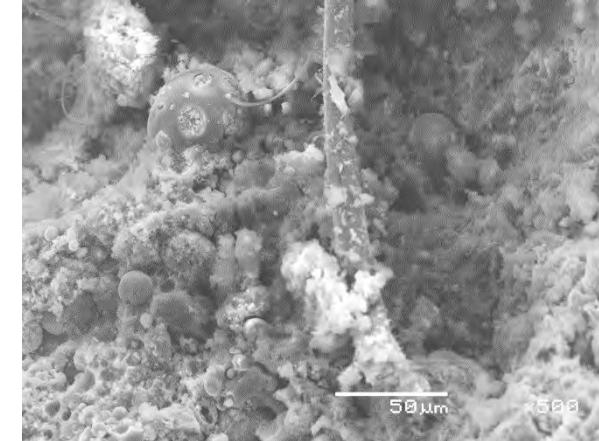
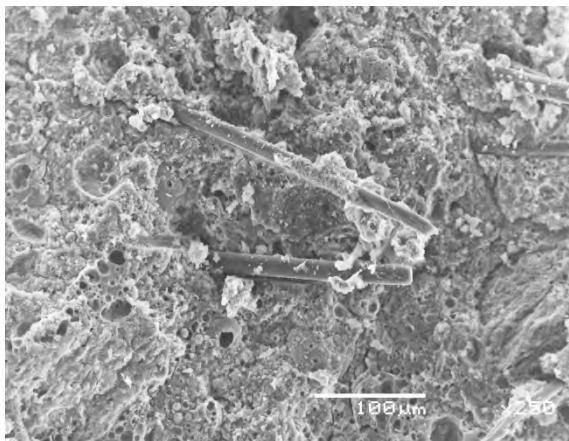
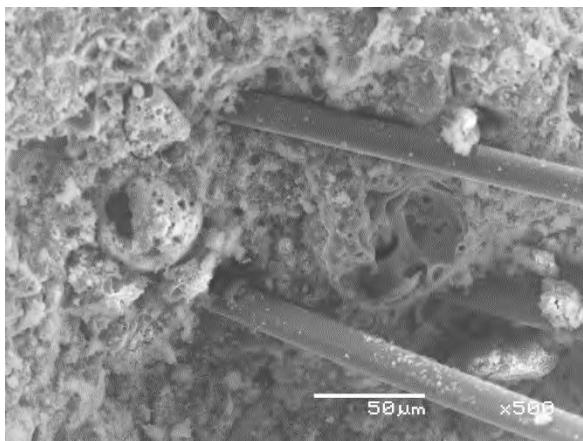
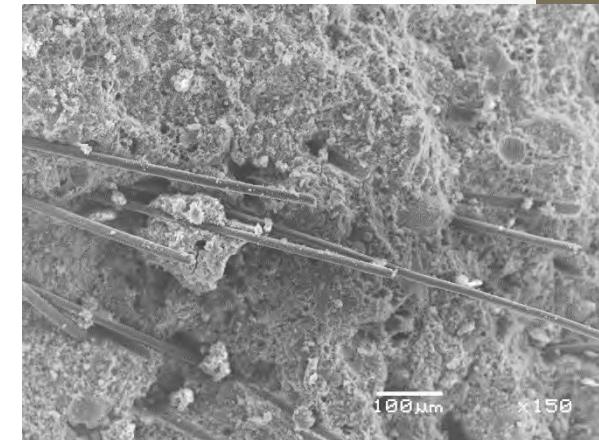
1 wt. % PVA A8 4 mm fibres



1,5 wt. % fibres

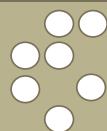
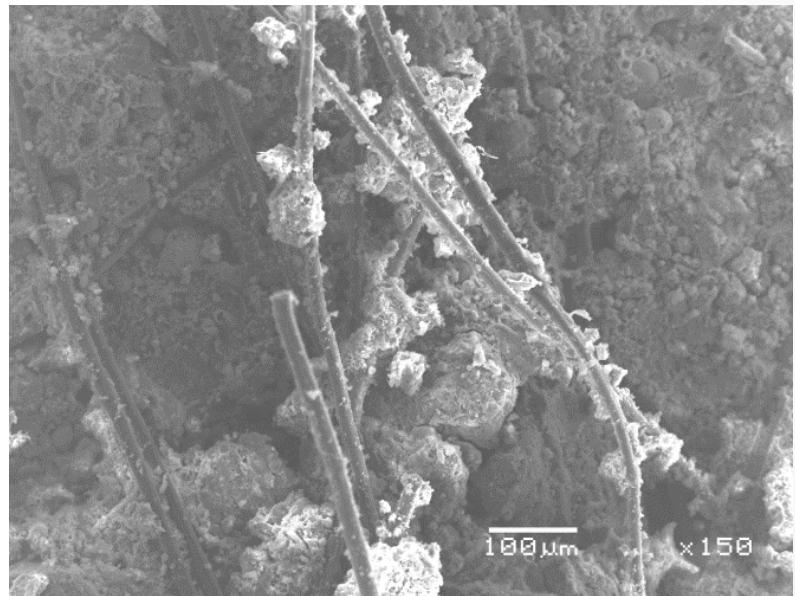


2 wt. % fibres

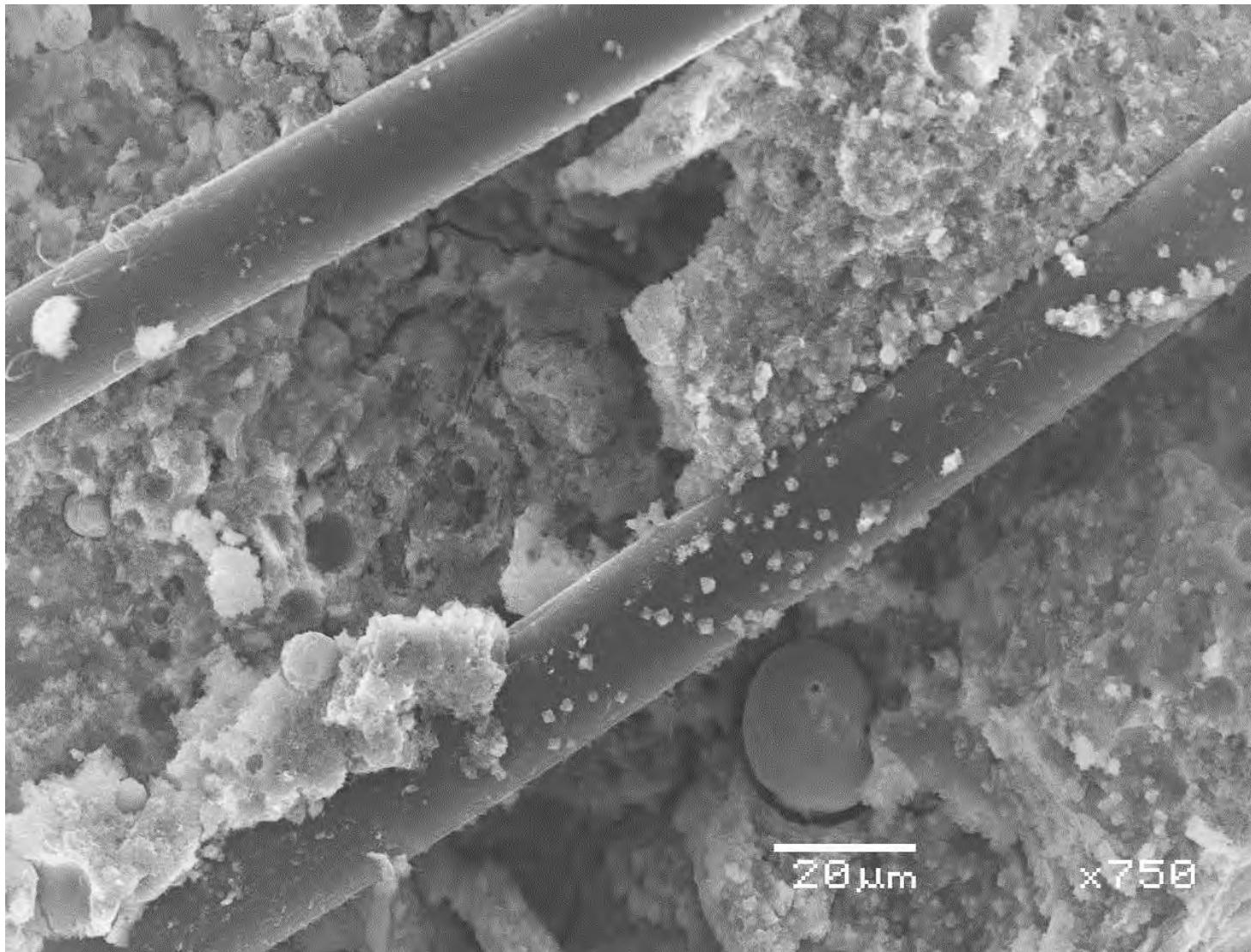


Fracture surfaces

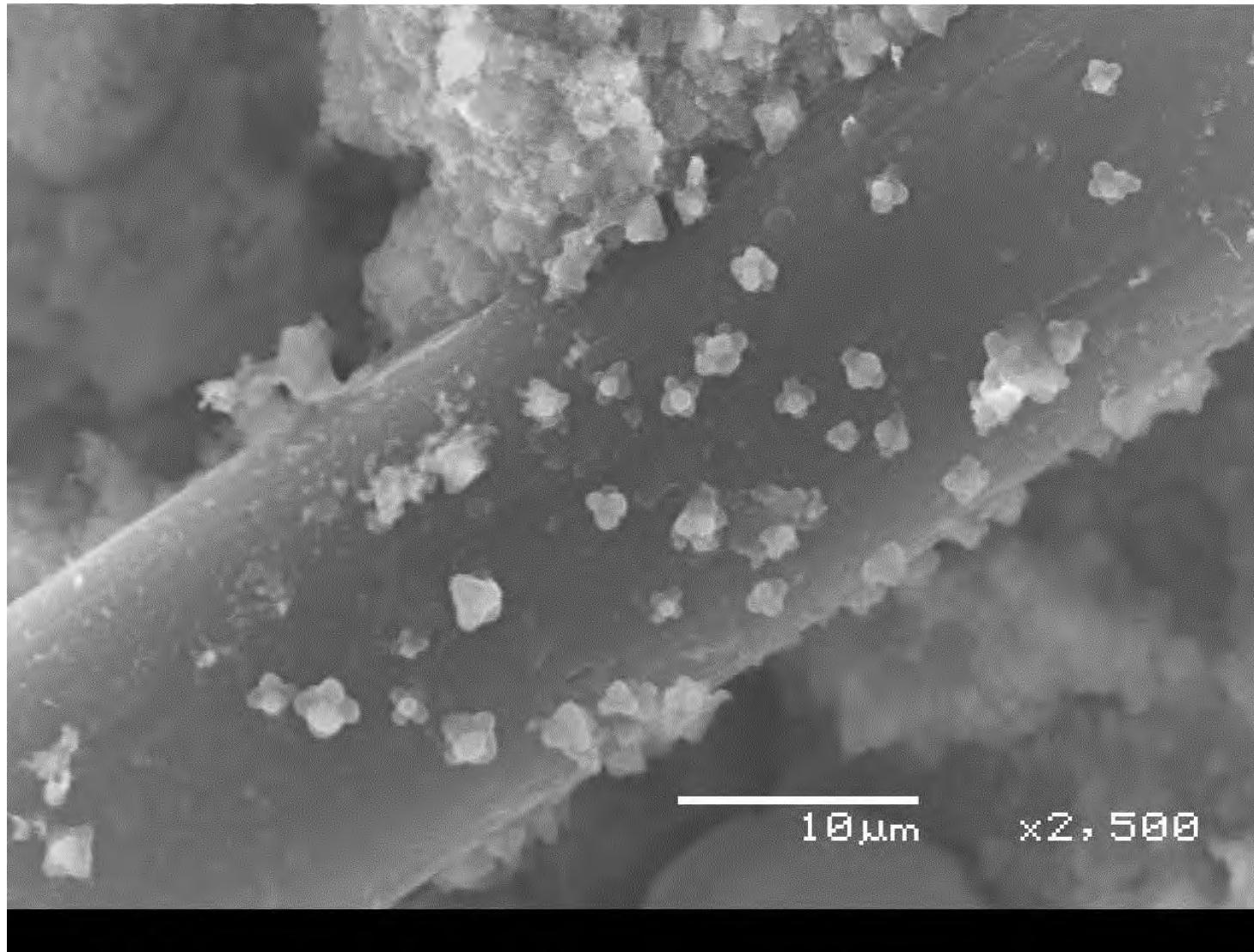
1 wt. % PVA A8 4 mm fibres



Fracture Surfaces

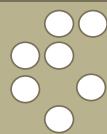


Fracture Surfaces

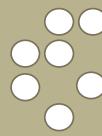
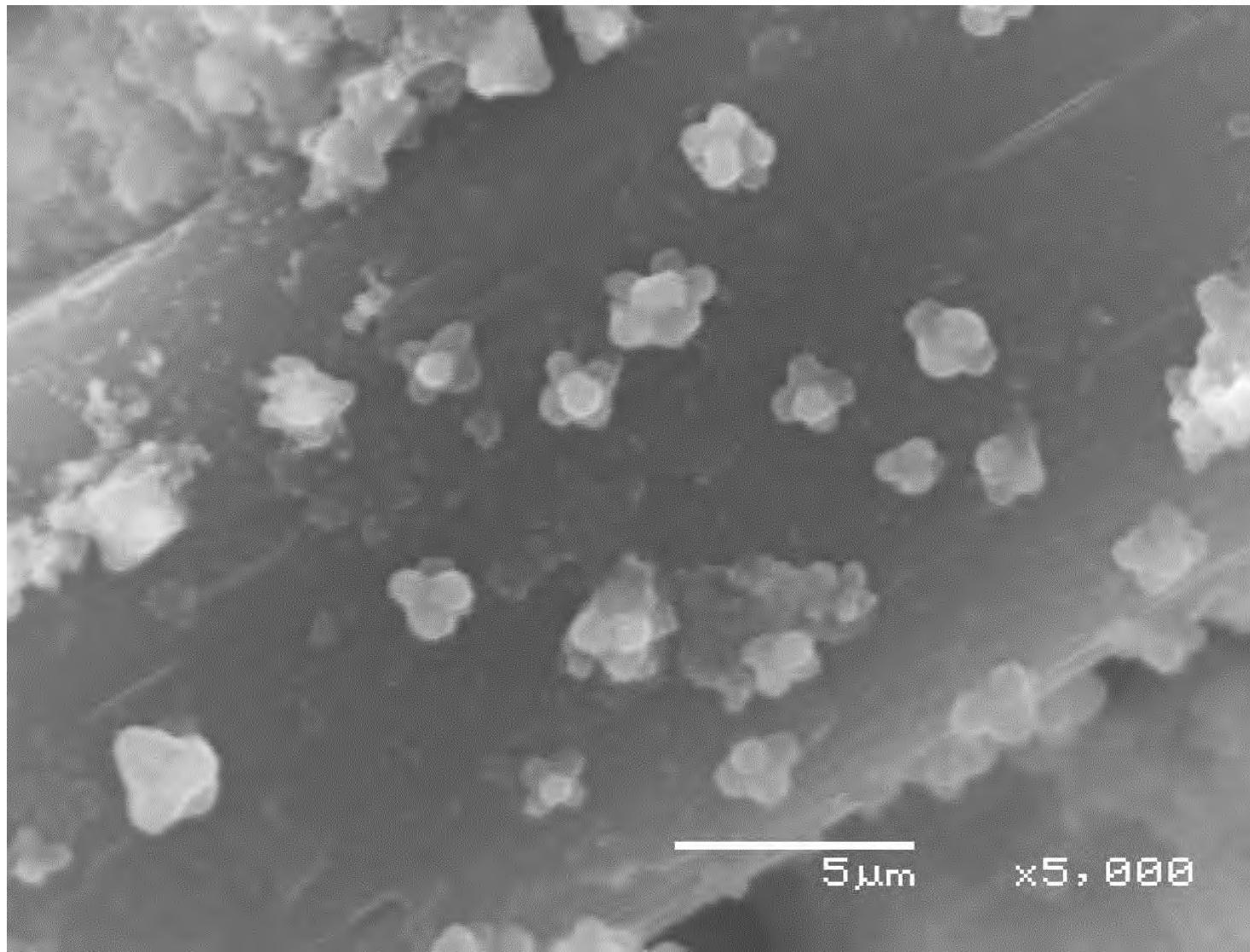


10 μm

$\times 2,500$

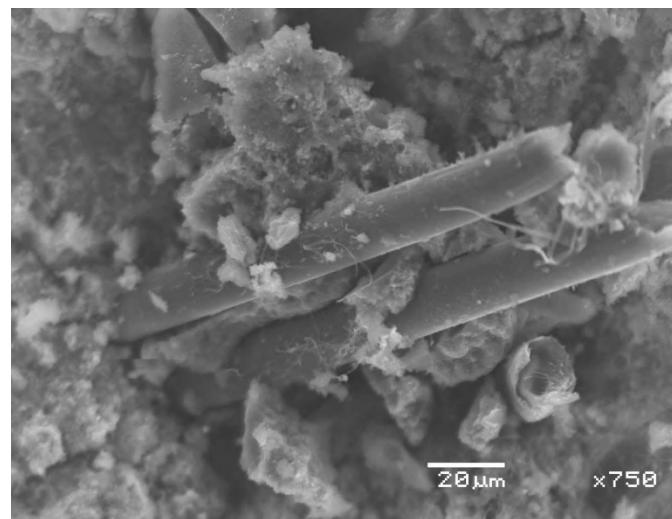
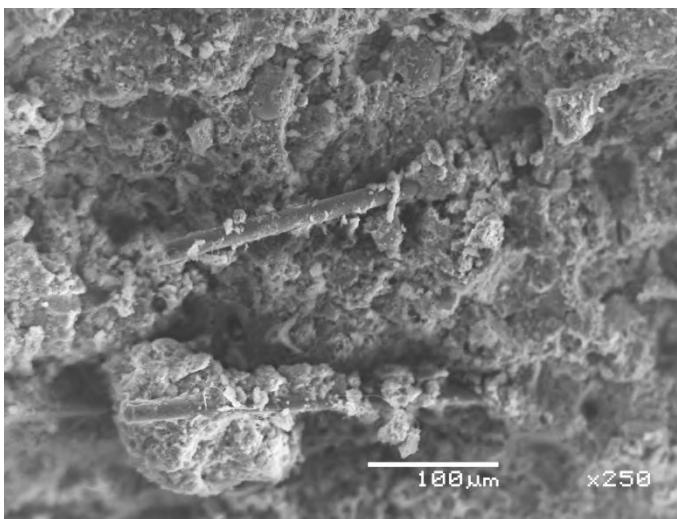
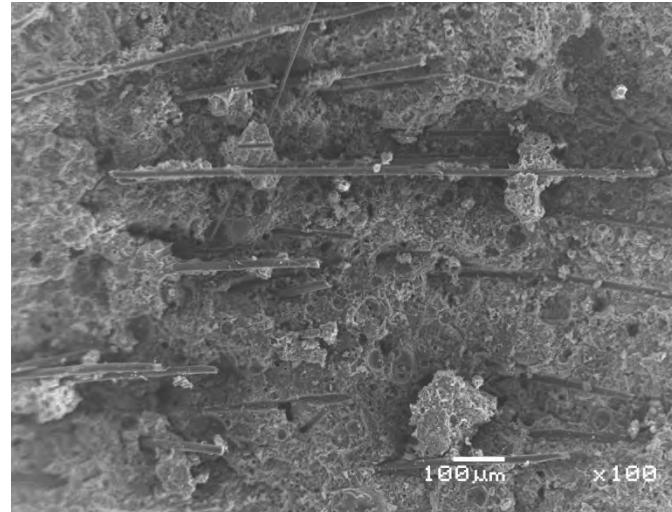
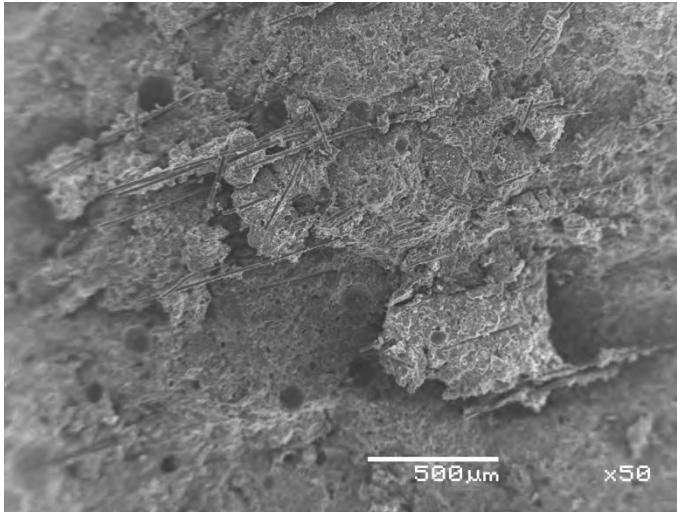


Fracture Surfaces



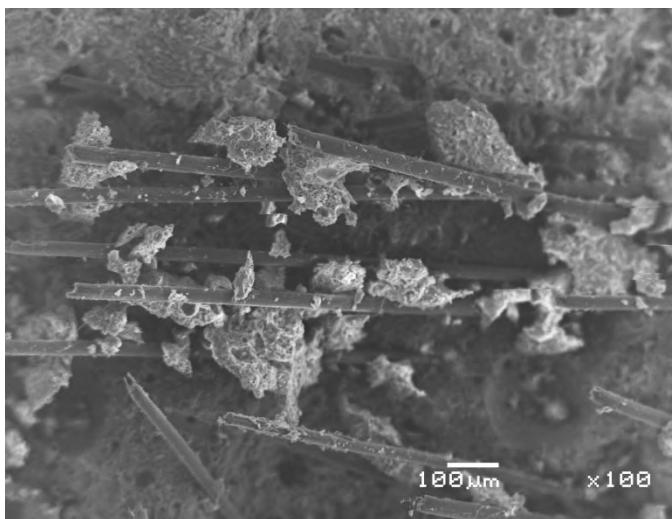
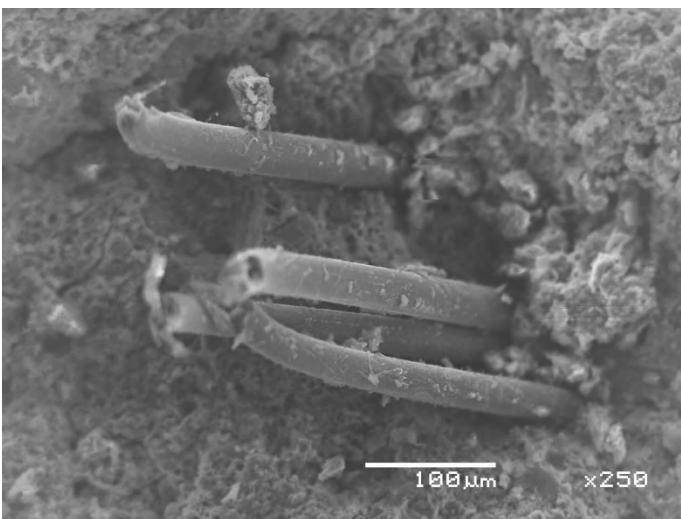
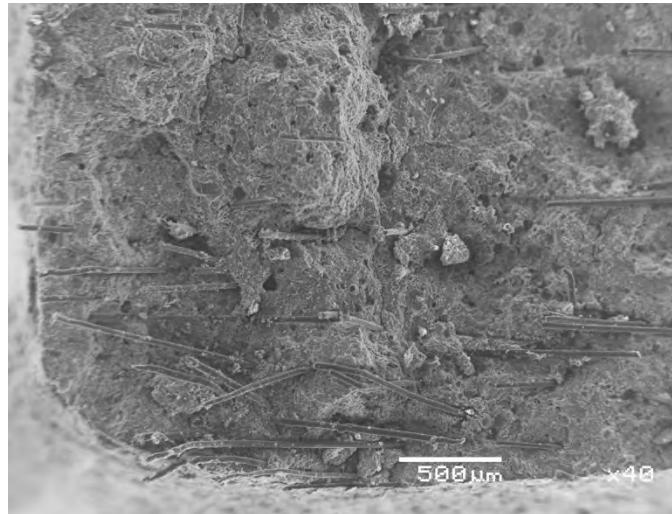
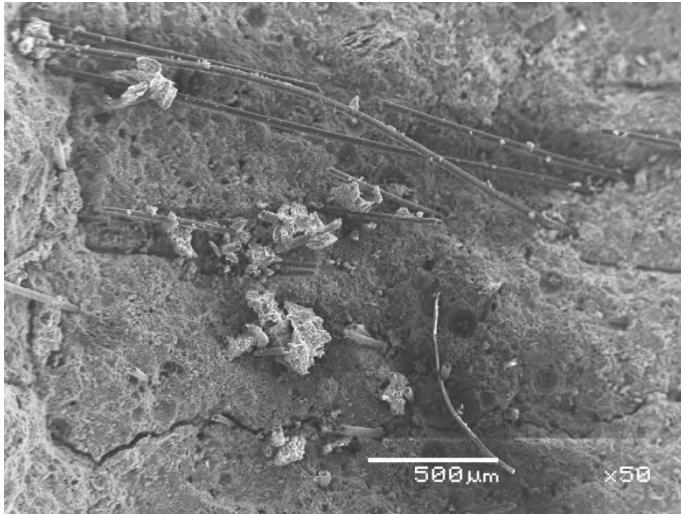
Fracture Surfaces

1 wt. % PVA A20 fibres – 28 days



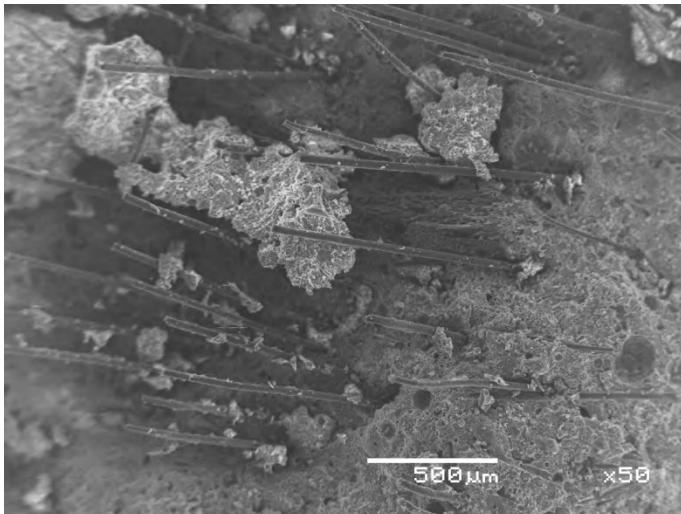
Fracture Surfaces

1 wt. % PVA A70 fibres – 7 days

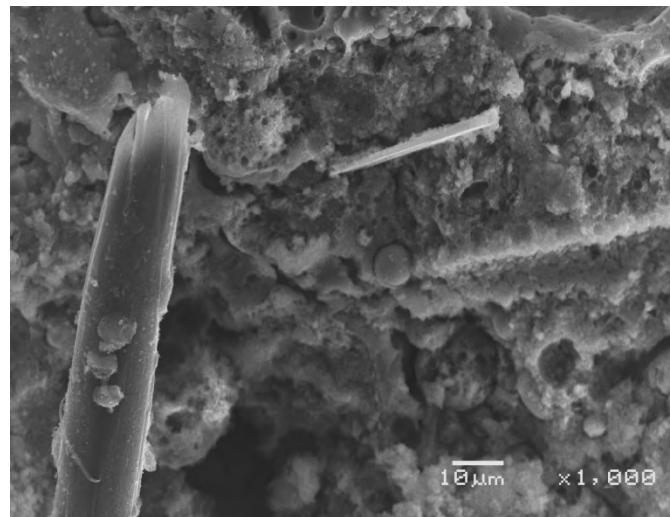
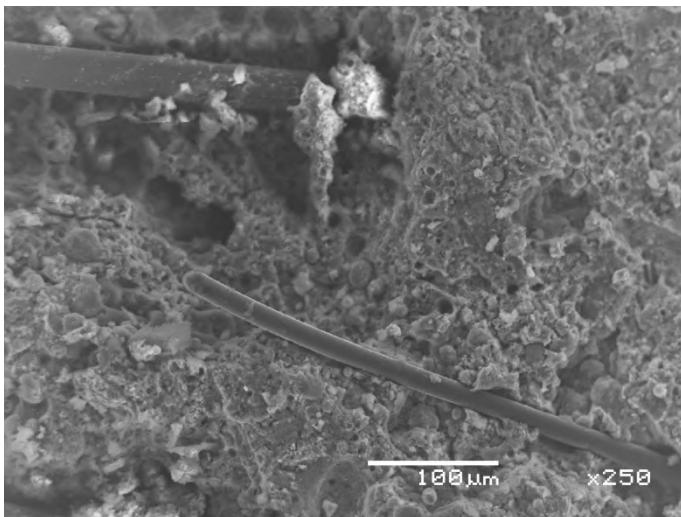
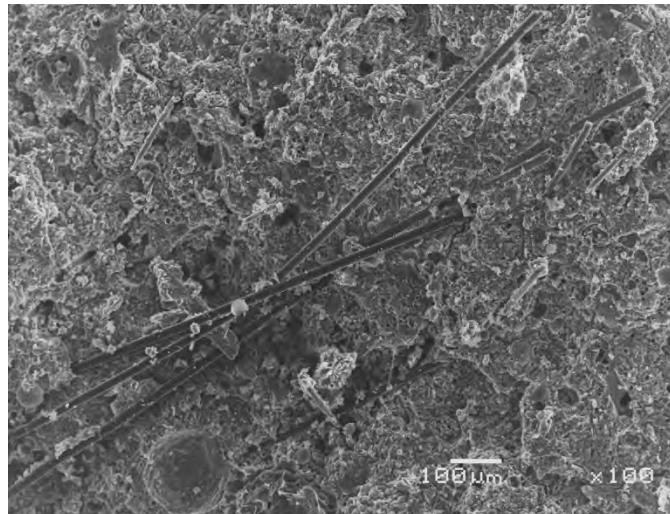


Fracture Surfaces

1 wt. % PVA A70 + Cf

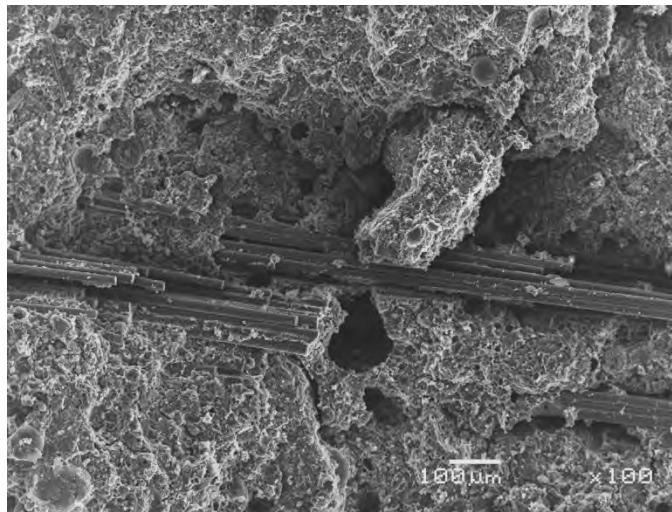
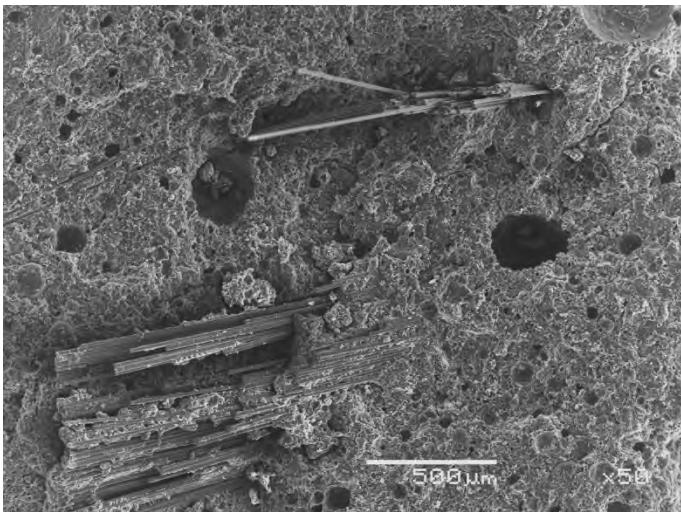
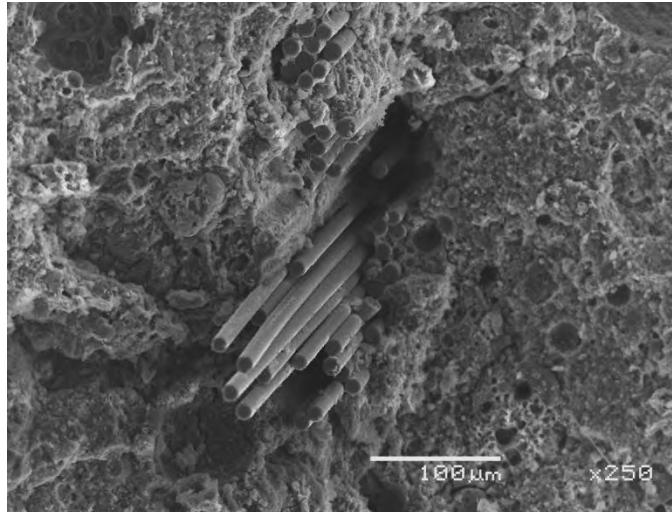
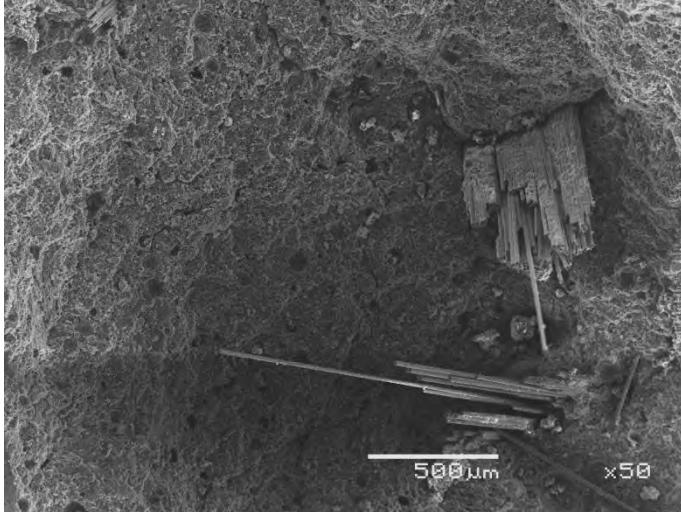


1 wt. % PVA A70 + Wol



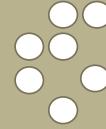
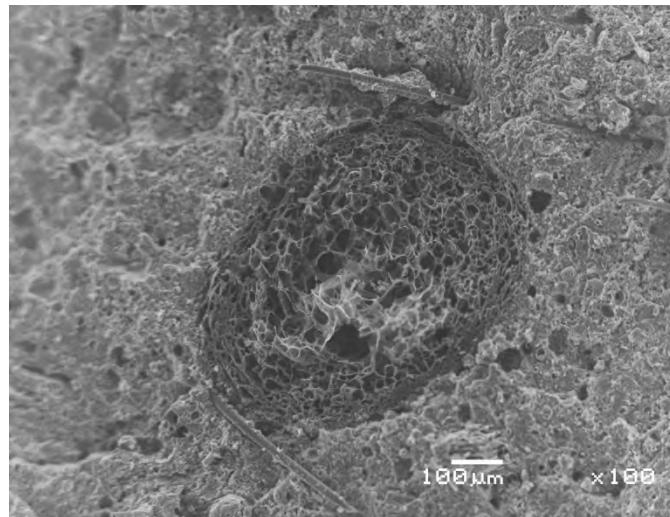
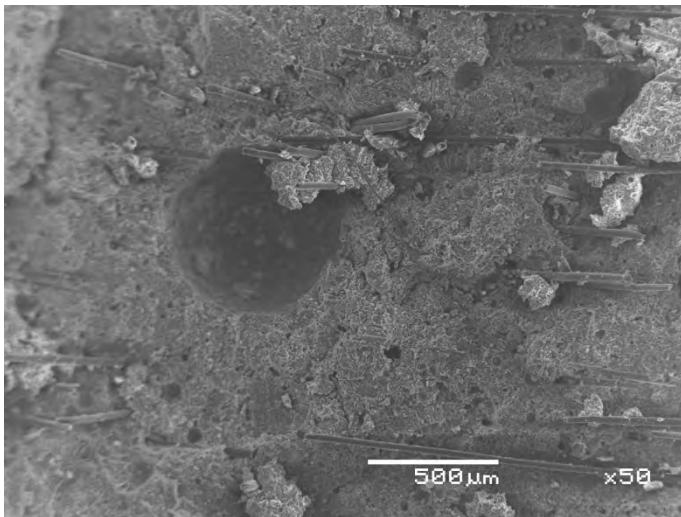
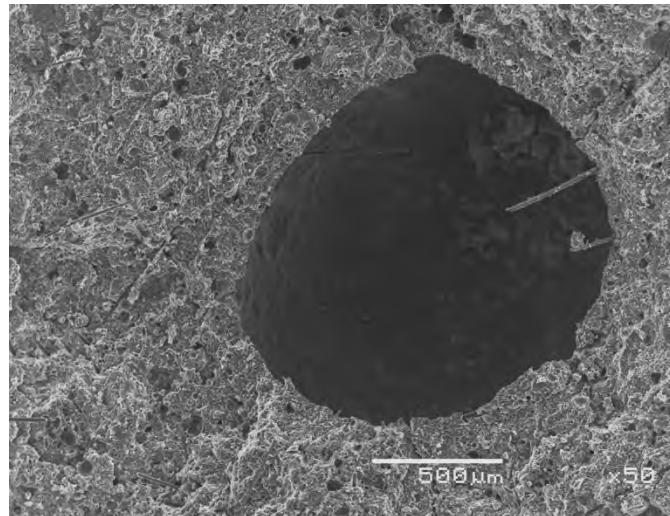
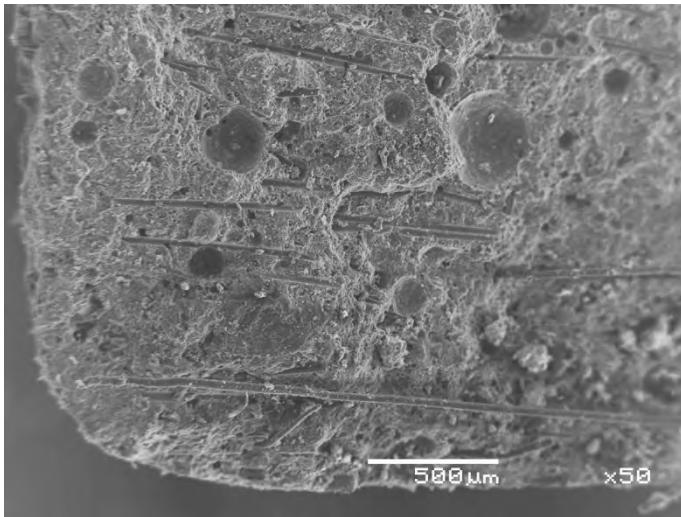
Fracture Surfaces

2 wt. % glass fibers (CemFill)



Strength determining flaws

1 wt. % PVA A70 fibres



Conclusions

- The research indicated, that fibers reinforced composites can be prepared using fly-ash geopolymers as a matrix phase by mechanical mixing of the constituents
- The flexural strength of the composites above 10 MPa can easily be reached,
- Best results were obtained using the PVA A70 6 mm fibers
- As observed from the SEM analysis of fracture surfaces, the bonding between the matrix and the PVA fibers is good
- The density of the composite with 1 wt.% PVA A70 fibers is $2,35 \text{ g/cm}^3$

