IPPT PAN
Institute of Fundamental Technological Research
Polish Academy of Sciences

Voids characterization in air entrained concrete specimens using optical microscopy and mercury intrusion porosimetry

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RADCON meeting, 25-26 September, 2019, Warsaw, Poland

Voids in concrete

- Interlayer space in C-S-H (5 to 25 Å)
- Capillary voids (may range from 10 to 50 nm or may be as large as 3 to 5 μm in low or high water-cement ratio pastes, irregular in shape)
- Air voids (formed by entrapped air during concrete mixing or by entrained air by using special admixtures, usually spherical, entrained air voids usually range from 10 to 500 μm)

Depending on the size and their distribution, the voids in the hydrated cement paste are considered as capable of adversely influencing the strength, promoting ingress of destructive media or enhancing the resistance to freeze-thaw damage

Air voids distribution

The distance between the air voids is important microstructural parameter

Frost: volume expansion during water-ice phase transition

↓

The distance between the nearest air voids should be small enough not to allow significant pressure increase

↓

Pressure release in air voids = frost resistance

Purpose of research

The investigation is aimed to get a quantitative description of voids in specimens of air-entrained concrete by means of complementary experimental techniques, like optical microscopy, x-ray microtomography and neutron imaging

Purpose of research

Compare three measuring techniques:

- neutron imaging → Budapest Neutron Centre, Hungary
- X-ray microtomography → Yonsei University, Korea
- **optical analysis → IPPT PAN, Poland**

Parameters of the air-void system:

(A) the total content of voids,

(B) the air-void size distribution,

(C) the void-to-void proximity (the distribution of the distance between the air voids)

Concrete mix design

Mix component		Mass content [kg/m³]			Density [kg/dm³]	Volume [liters]		
		S61	W-P-2	GWB19		S61	W-P-2	GWB19
Portland Cement CEM I 42.5R		420	360	425	3.1	135.5	116.1	137.1
Water		165	144	166	1	165.0	144.0	166.0
Mineral aggregate	Quartz sand 0/2 mm	579	551	525	2.65	218.5	207.9	198.1
	Amphibolite 2/5 mm	524			2.91	180.1		
	Amphibolite 5/8 mm	687			2.91	236.1		
	Amphibolite 2/8 mm		228		2.9		78.6	
	Amphibolite 8/16 mm		475		2.9		163.8	
	Quartzite 16/32 mm		645		2.65		243.4	
	Gabbro 2/4 mm			326	2.94			110.9
	Gabbro 4/8 mm			991	2.94			337.1
Admixtures	Plasticizer	1.89	1.8	2.89	1.04	1.8	1.7	2.8
	Air entraining agent	0.5	0.58	0.77	1.05	0.5	0.6	0.7

Concrete mix design

The content of concrete components based on mix design data

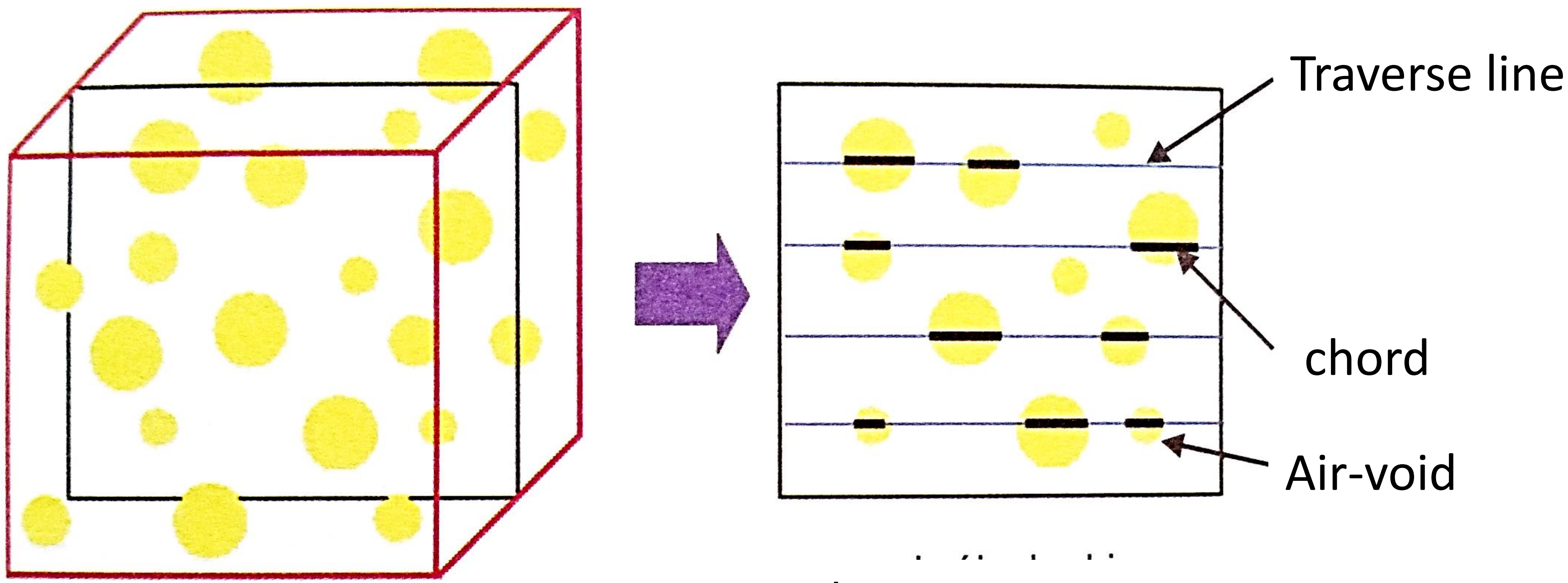
Concrete component	Relative volume content		
	S61	W-P-2	GWB19
Hardened cement paste	0.300	0.260	0.303
Air voids (entrained and entrapped)	0.066	0.016	0.042
Fine aggregate (quartz sand)	0.218	0.208	0.198
Coarse aggregate	0.415	0.516	0.457

Testing methods

- the determination of air void characteristics according to European standard EN 480-11 with the use of the computerized automatic image analysis system,
- the determination of pore size distribution using mercury intrusion porosimetry,
- the determination of the rate of water absorption following ASTM C1585 standard to provide a physical measure of voids connectivity in concrete,
- the determination of the distance between the air voids using image analysis.

Linear traverse method according to EN 480-11

Linear traverse method EN 480-11

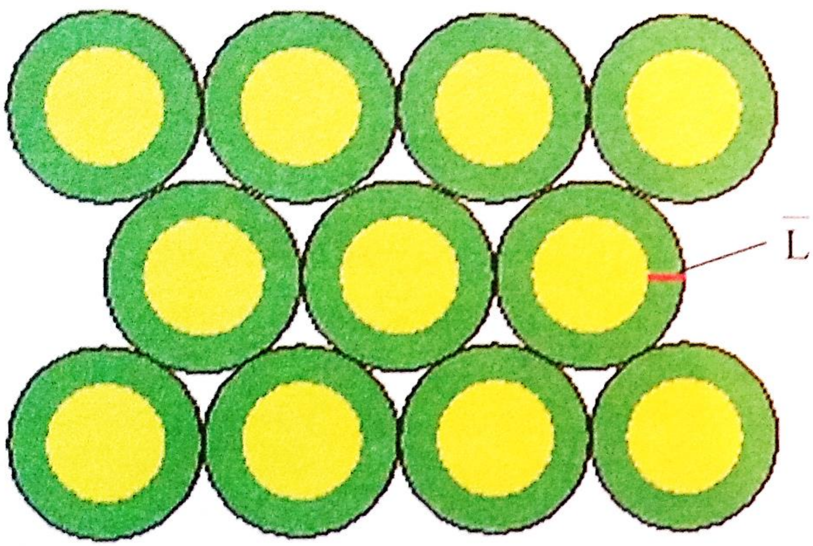


Plane cross-section

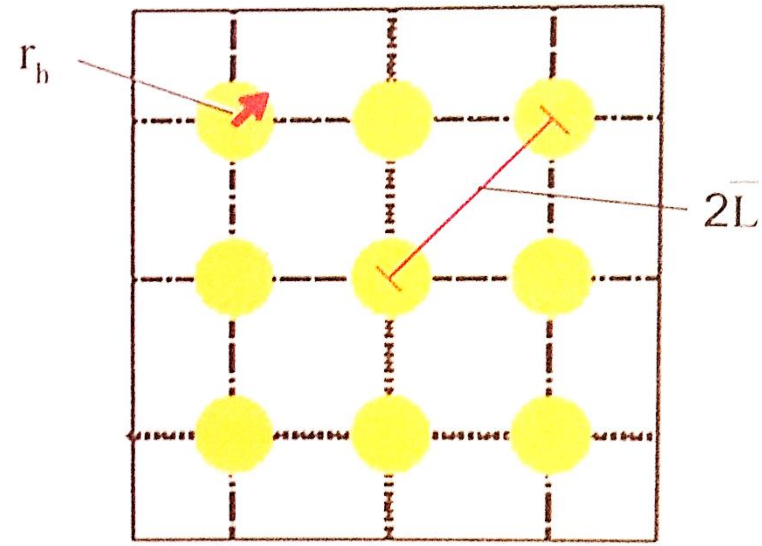
M.A. Glinicki, Trwałość betonu w nawierzchniach drogowych, Instytut Badawczy Dróg i Mostów, Warszawa 2011

Calculation of air void spacing factor L according to Powers

R ≤ 4.342



R > 4.342



$$\overline{L} = \frac{P \cdot T_{tot}}{400 \cdot N}$$

$$\overline{L} = \frac{3}{\alpha}\left[1{,}4\left(1+R\right)^{1/3} - 1\right]$$

R – paste-air ratio

M.A. Glinicki, Trwałość betonu w nawierzchniach drogowych, Instytut Badawczy Dróg i Mostów, Warszawa 2011

Microstructure parameters according to EN 480-11

Total air content:

$$A = \frac{T_a \cdot 100}{T_{tot}} \quad [\%]$$

Specific surface:

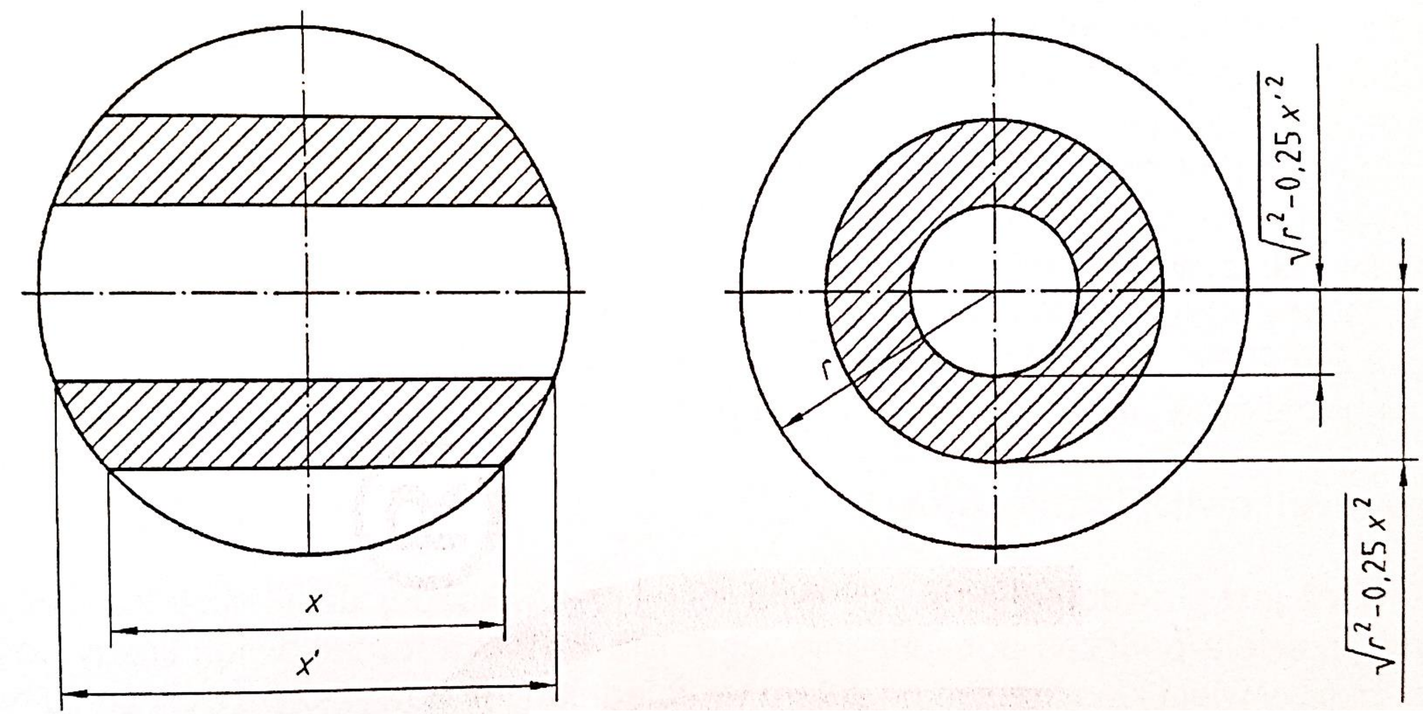
$$\alpha = \frac{4 \cdot N}{T_a} \quad [mm^{-1}]$$

Micropores content (<300 µm):

$$A_{300} \quad [\%]$$

(different stastistical model)

Theoretical basis of A_{300} calculations



side view (along the measuring line)

cross section (perpendicular to the measuring line)

The probability of the intersection of the air void with a radius r:

$$\frac{\pi\cdot(y' + y)\cdot(y' - y + 5)}{4\cdot 10^6}$$

Theoretical basis of A_{300} calculations

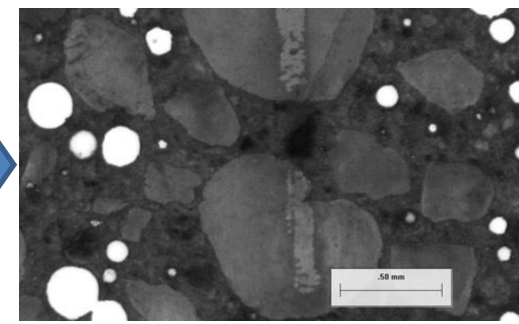
	Total length of the traverse line, Ttot=		1207,62	mm					
Column	1	2	3	4	5	6	7	8	9	10
	Air voids class	Class range	Numeber of chords in class	Frequency of chords	Share of counted air voids	Potential number of chords	Number of air voids in class	Singular air void volume	Air content	Cumulated air content
		µm		mm^{-1}	mm^2	mm^{-3}	mm^{-3}	mm^3	%	%
	1	0 do 10	42	0.034779177	0.0001178	295.239197	36.18315441	0.000000524	0.0019	0.00
	2	15 do 20	86	0.071214506	0.0002749	259.056043	140.2118519	0.000004190	0.0587	0.06
	3	25 do 30	62	0.051340691	0.000432	118.844191	-0.65738887	0.000014100	-0.0009	0.06
	4	35 do 40	85	0.070386431	0.000589	119.50158	45.14014201	0.000033500	0.1512	0.21
	5	45 do 50	67	0.055481069	0.0007461	74.3614378	39.52211673	0.000065400	0.2585	0.47
	6	55 do 60	38	0.031466875	0.0009032	34.8393211	13.39223439	0.000113000	0.1513	0.62
	7	65 do 80	59	0.048856464	0.002278	21.4470867	9.479028412	0.000268000	0.2540	0.87
	8	85 do 100	42	0.034779177	0.002906	11.9680583	7.516038665	0.000524000	0.3938	1.27
	9	105 do 120	19	0.015733437	0.003534	4.45201964	1.468321635	0.000905000	0.1329	1.40
	10	125 do 140	15	0.012421135	0.004163	2.98369801	1.255299648	0.001440000	0.1808	1.58
	11	145 do 160	10	0.008280757	0.004791	1.72839836	0.200301563	0.002140000	0.0429	1.63
	12	165 do 180	10	0.008280757	0.005419	1.5280968	0.980391488	0.003050000	0.2990	1.92
	13	185 do 200	4	0.003312303	0.0060476	0.54770531	-0.44459626	0.004190000	-0.1863	1.74
	14	205 do 220	8	0.006624605	0.006676	0.99230156	0.652182865	0.005580000	[illegible]	2.10
	15	225 do 240	3	0.002484227	0.007304	0.3401187	-0.49495066	0.007240000	-0.3583	1.74
	16	245 do 260	8	0.006624605	0.007933	0.83506936	0.254710298	0.009200000	[illegible]	1.98
	17	265 do 280	6	0.004968454	0.008561	0.58035906	0.49024309	0.011500000	0.5638	2.54
	18	285 do 300	1	0.000828076	0.009189	0.09011597	0.025724396	0.014100000	0.0363	2.58
	19	305 do 350	2	0.001656151	0.02572	0.06439157	-0.07525019	0.022400000	-0.1686	2.41
	20	355 do 400	5	0.004140378	0.02965	0.13964176	0.065662401	0.033500000	0.2200	2.63
	21	405 do 450	3	0.002484227	0.03358	0.07397936	0.029815327	0.047700000	0.1422	2.77
	22	455 do 500	2	0.001656151	0.0375	0.04416403	0.030152602	0.065400000	0.1972	2.97
	23	505 do 1000	10	0.008280757	0.591	0.01401143	0.012327838	0.524000000	0.6460	3.61
	24	1005 do 1500	2	0.001656151	0.9837	0.00168359	0.001081795	1.770000000	0.1915	3.81
	25	1505 do 2000	1	0.000828076	1.376	0.0006018	0.000601799	4.190000000	0.2522	4.06
	26	2005 do 2500	0	0	1.769	0	0	8.180000000	0.0000	4.06
	27	2505 do 3000	0	0	2.162	0	-0.00030101	14.10000000	-0.4244	[illegible]
	28	3005 do 4000	2	0.001656151	5.502	0.00030101		33.500000000	0.0000	3.63

Total air content A

Zawartosc zaczynu cementowego w betonie P=	27	%
Calkowita dlugosc cieciw przypadajaca na pory T_a=	55.288	mm
Calkowita zawartosc powietrza A=	4.58	%
Calkowita liczba mierzonych cieciw N=	592	
Powierzchnia wlasciwa porow α=	**42.83**	mm^{-1}
Stosunek zaczyn/powietrze R=	5.897	
Wskaznik rozmieszczenia L=	**0.12**	mm
Zawartosc mikroporow A300=	2.58	%

Optical microscopy with digital image analysis

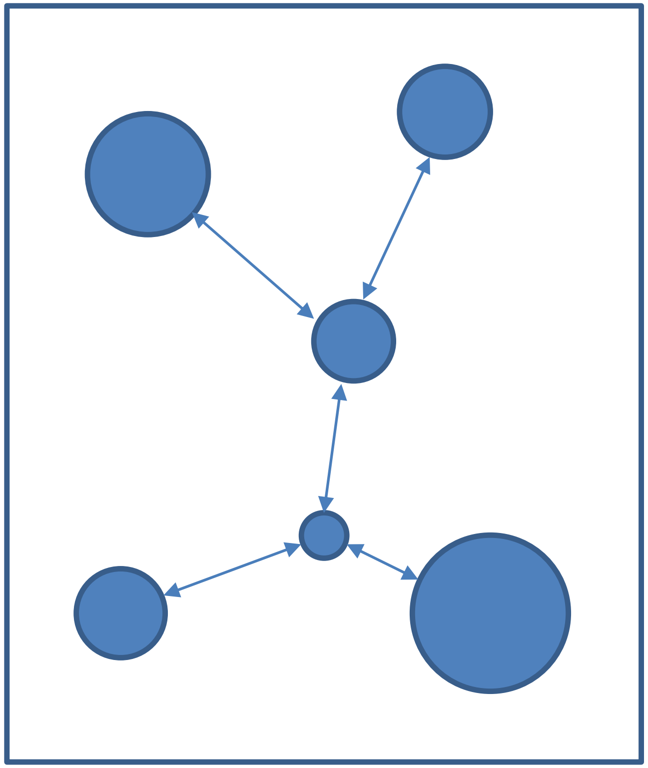
100 mm

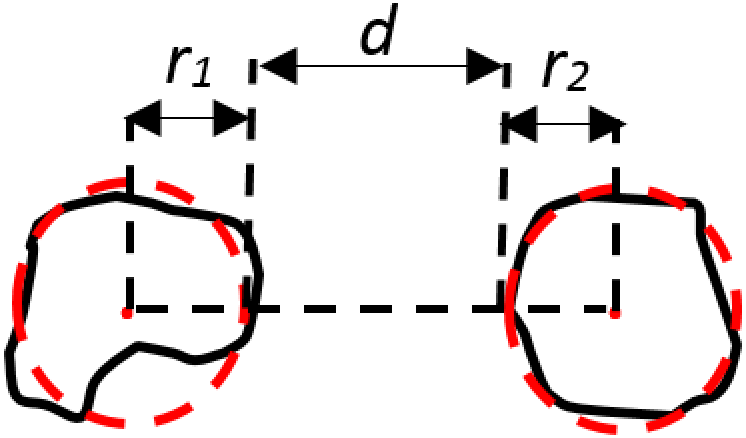


Scale bar = 0.5 mm

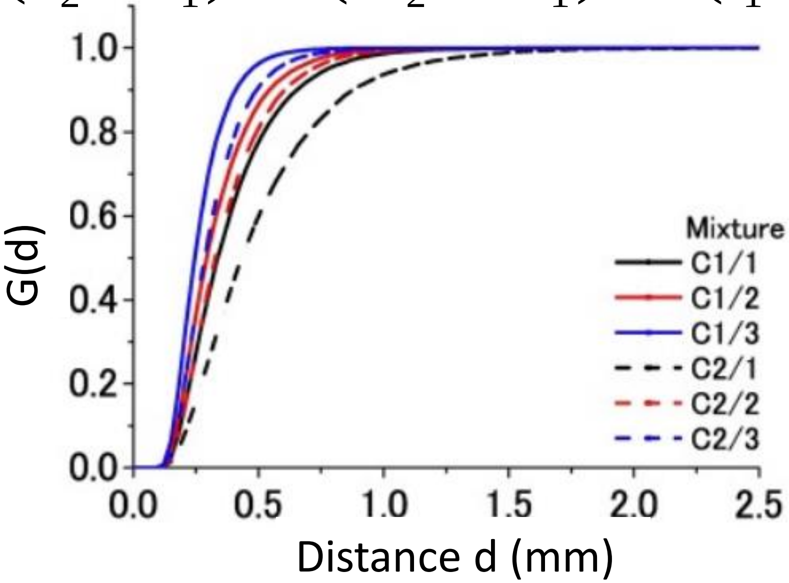
Distance between the air voids using image analysis

Nearest-Neighbour Spacing Distribution



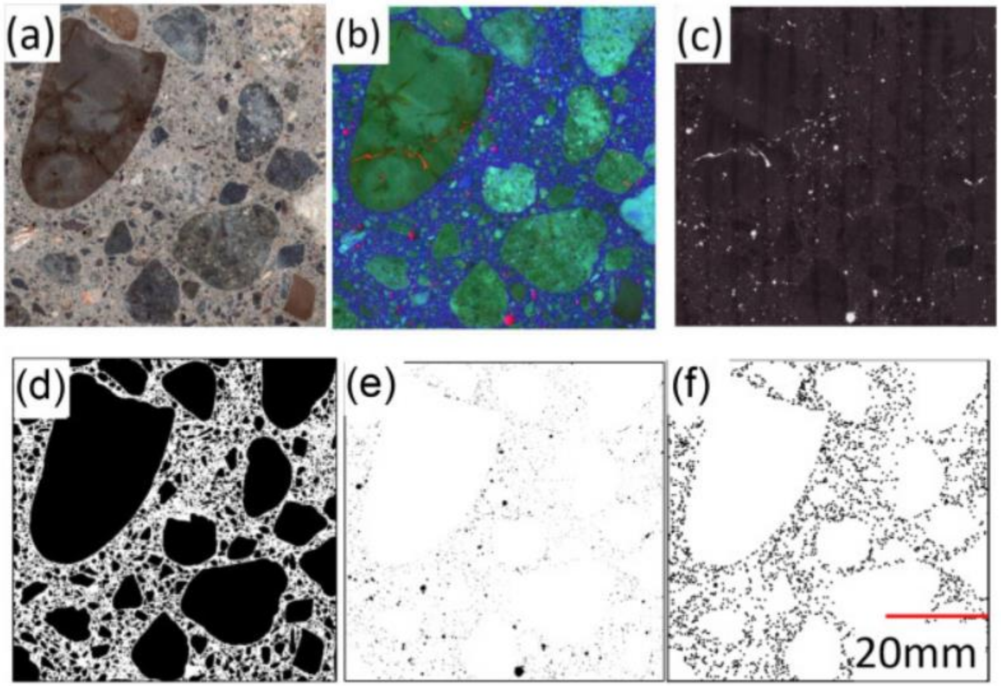


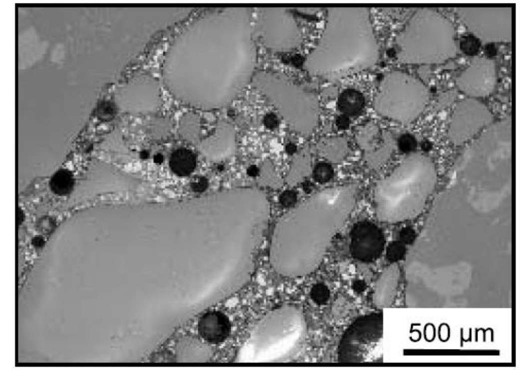
$$d = \sqrt{(Y_2 - Y_1)^2 + (X_2 - X_1)^2} - (r_1 + r_2)$$

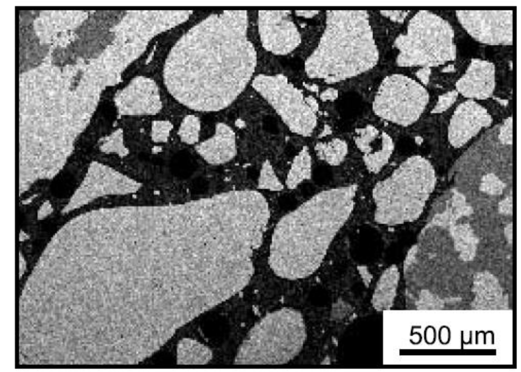


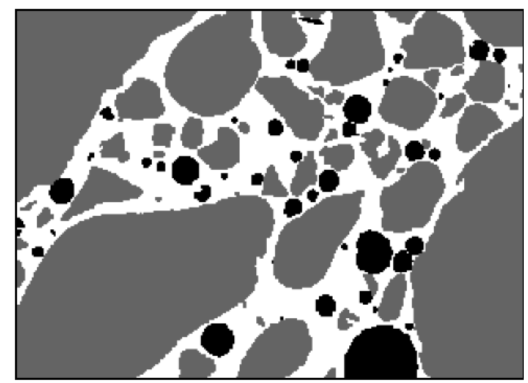
T.Murotani, S.Igarashi, H.Koto, Distribution analysis and modeling of air voids in concrete as spatial point processes, Cement and Concrete Research 115, 124-132, 2019

Sample preparation







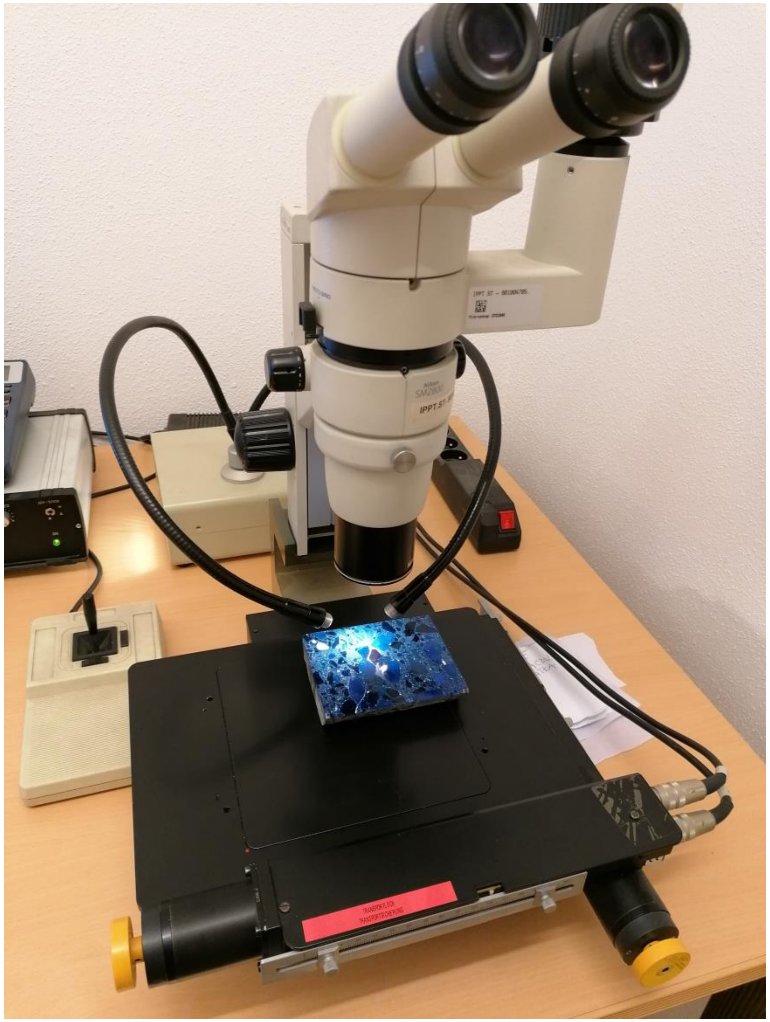


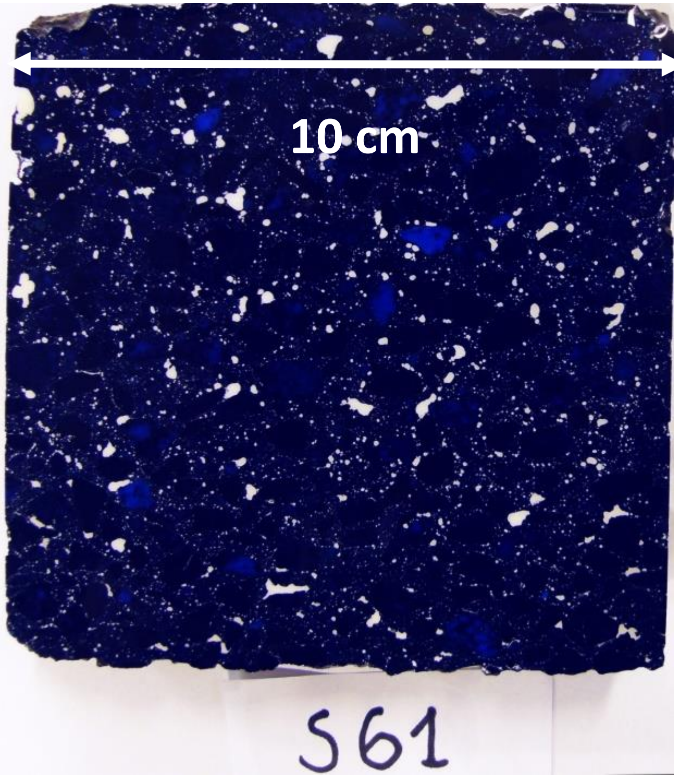
Extraction of a point pattern for the air voids:
(a) original scanned image, (b) segmentation and coloring of the different phases, (c) scanned air voids, (d) segmented aggregate particles, (e) inversion of image (c), (f) point pattern of image (e).

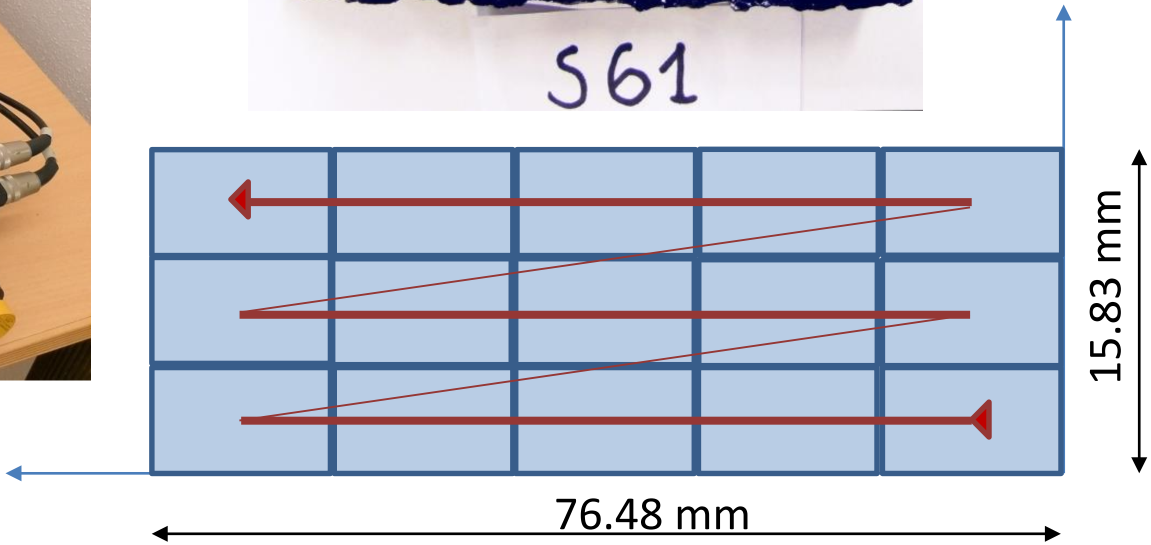
T.Murotani, S.Igarashi, H.Koto, Distribution analysis and modeling of air voids in concrete as spatial point processes, Cement and Concrete Research 115, 124-132, 2019

Anne Sophie Dequiedt, Michel Coster, Liliane Chermant, Jean-Louis Chermant, Distances between air-voids in concrete by automatic methods, Cement and Concrete Composites, 23, 2–3, 2001, 247-254

Image acquisition







ImageJ

Algorithm

In a close packed configuration of particles/fibers having a circular cross section in 2D space there are 6 immediate neighbors surrounding each particle. In randomly packed systems, coordination number depends on the visual perception and can be lower or higher. Estimation of particle spacing of a particle with its neighboring particles is performed as follows:

1. The centroid coordinates of each particle (X,Y) is derived from the result table of the built-in Analyse Particles plugin.
2. A circle is fit on each particle with the center (X,Y) and radius r.
3. The spacing (wall thickness) between a pair of particles (d) is calculated as:

$$d = \sqrt{(Y_2 - Y_1)^2 + (X_2 - X_1)^2} - (r_1 + r_2)$$

4. The distances of each particle with all the other particles is stored in an array and sorted.
5. Results are shown in a new result table, which contain the distance of the closest neighbor to each particle.

Haeri, M. and Haeri, M., 2015. ImageJ Plugin for Analysis of Porous Scaffolds used in Tissue Engineering. *Journal of Open Research Software*, 3(1), p.e1.

ImageJ – results of analysis

Results

File Edit Font Results

	Label	Area	X	Y	Major	Minor	Angle
1	WP_1_M-1.tif	0.001527	4.632939	0.013576	0.068386	0.028424	18.172488
2	WP_1_M-1.tif	0.003008	13.801363	0.012064	0.129120	0.029666	0.600520
3	WP_1_M-1.tif	0.067852	17.925548	0.147494	0.460726	0.187514	138.209762
4	WP_1_M-1.tif	0.001553	20.541610	0.011364	0.074870	0.026405	4.114537
5	WP_1_M-1.tif	0.007955	21.344184	0.029602	0.141364	0.071648	178.551709
6	WP_1_M-1.tif	0.009569	23.322302	0.040848	0.129509	0.094075	1.801760
7	WP_1_M-1.tif	0.001583	24.073141	0.012000	0.068837	0.029287	179.563504
8	WP_1_M-1.tif	0.055937	25.323966	0.104793	0.297788	0.239166	3.482647
9	WP_1_M-1.tif	0.001940	25.520434	0.021527	0.051833	0.047661	174.546470
10	WP_1_M-1.tif	0.001397	30.025158	0.008239	0.087323	0.020365	1.348554
11	WP_1_M-1.tif	0.002966	49.065055	0.015293	0.101067	0.037365	0.093712
12	WP_1_M-1.tif	0.001720	49.542561	0.011027	0.080728	0.027135	179.683507
13	WP_1_M-1.tif	0.002470	62.807307	0.015406	0.082768	0.037991	1.203543
14	WP_1_M-1.tif	0.186958	76.300222	0.249063	0.560809	0.424462	98.720893
15	WP_1_M-1.tif	0.013284	22.288126	0.075928	0.141146	0.119832	150.164175
16	WP_1_M-1.tif	0.005625	3.476291	0.060311	0.088203	0.081193	163.066032
17	WP_1_M-1.tif	0.003188	2.555008	0.063140	0.073931	0.054905	102.564805
18	WP_1_M-1.tif	0.012006	26.262040	0.090939	0.125267	0.122027	12.220073
19	WP_1_M-1.tif	0.005145	23.045348	0.074981	0.150931	0.043402	15.920830
20	WP_1_M-1.tif	0.001056	24.599054	0.051672	0.038034	0.035364	35.830710
21	WP_1_M-1.tif	0.001711	6.289699	0.063758	0.055074	0.039557	162.116004
22	WP_1_M-1.tif	0.002616	4.397459	0.074438	0.061736	0.053955	20.174476

Distance Between Neighboring Particles.csv

File Edit Font

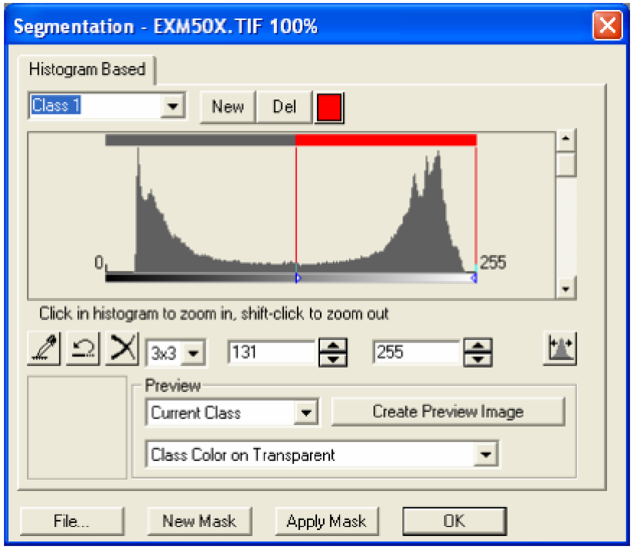
Average Distance From 3 Neighbors	Nearest Neighbor Distance
0.330913	0.197693
0.628554	0.404906
0.438894	0.267943
0.604283	0.524805
0.285243	0.139665
0.325844	0.226859
0.588860	0.487779
0.256761	0.213384
0.268293	0.190998
0.610418	0.555495
0.462182	0.279340
0.452339	0.421078
0.457255	0.319453
0.754071	0.574406
0.658529	0.564016
0.623938	0.363707
0.595467	0.538923
0.629419	0.441146
0.247640	0.092108

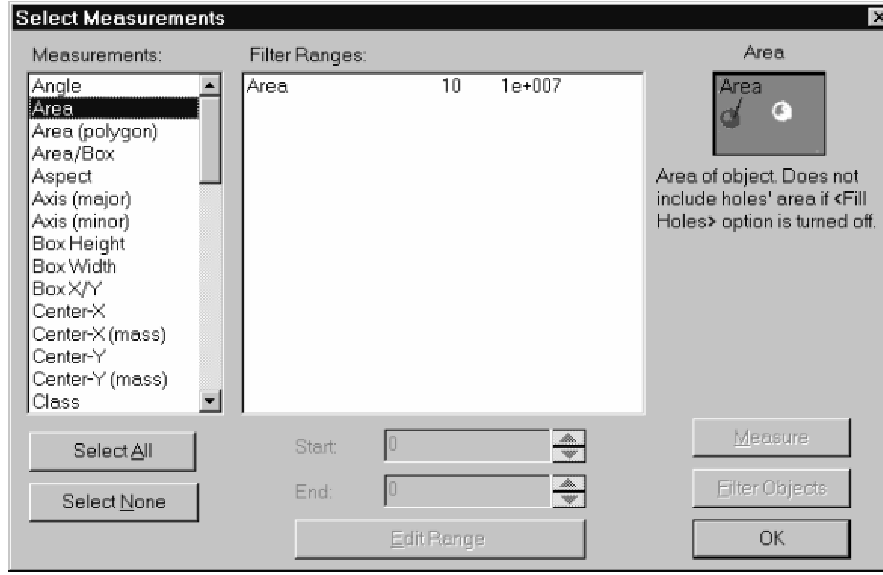
Summary

File Edit Font

Slice	Count	Total Area	Average Size	%Area	Major	Minor	Angle
WP_1_M-1.tif	1717	20.787243	0.012107	1.716453	0.109704	0.084885	91.692715

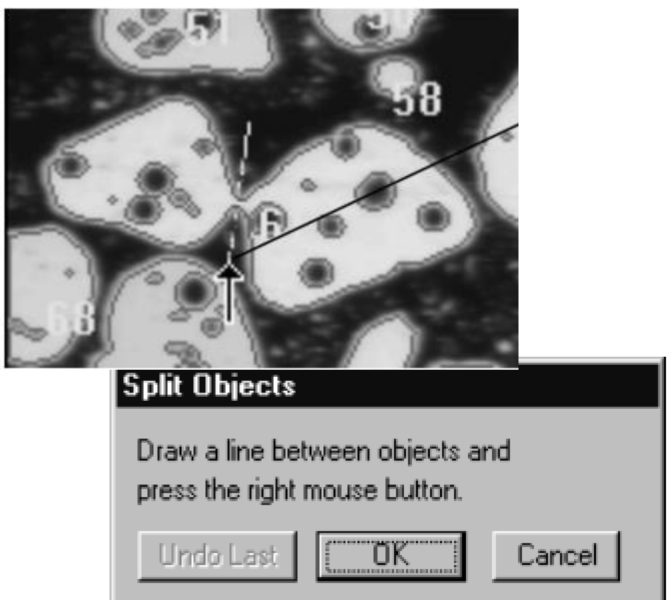
Image Pro Plus





Area, PerArea, Center-X, Center-Y, Diameter (min, max), Radius (min, max)

225 - 255

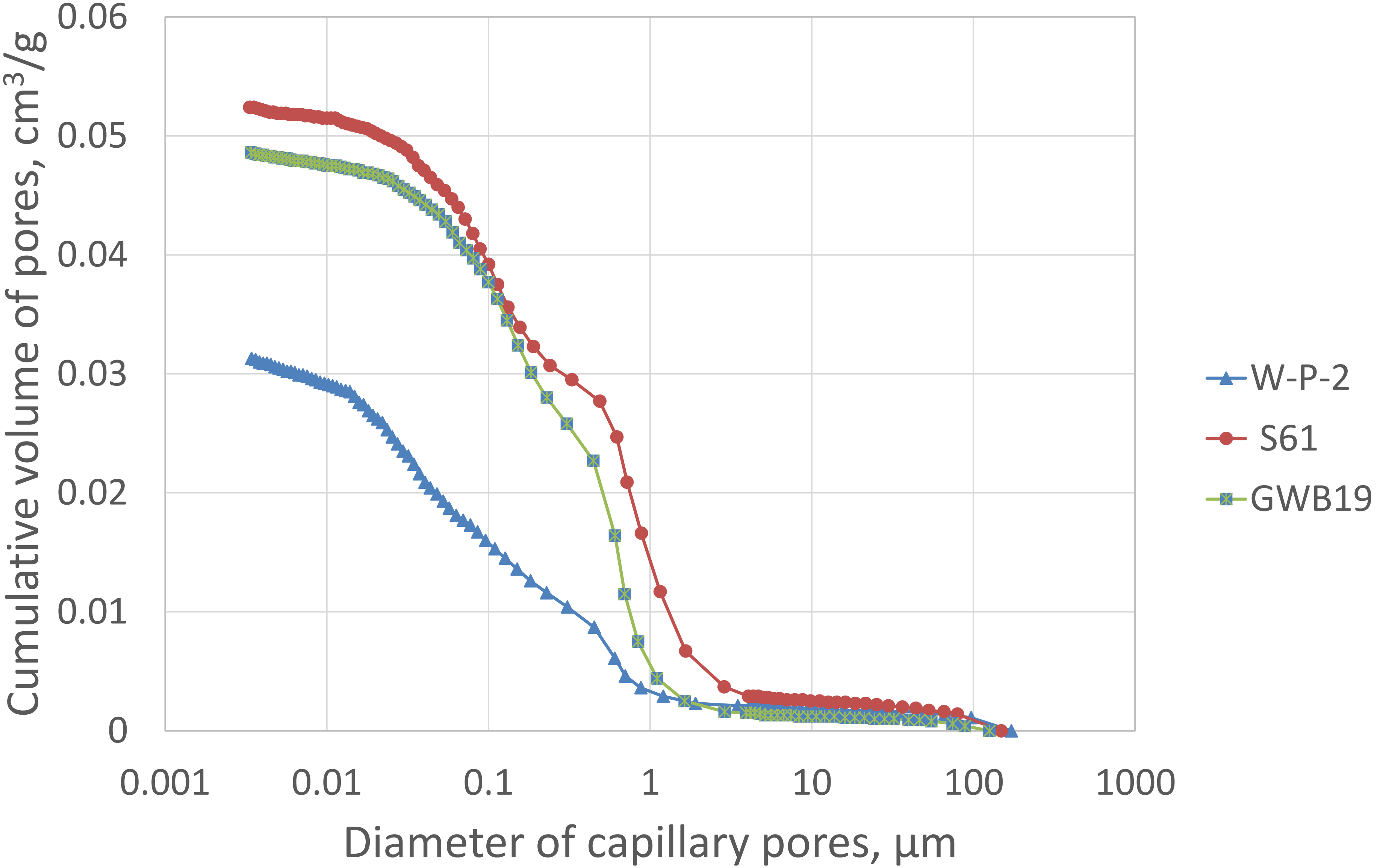


Manual spliting

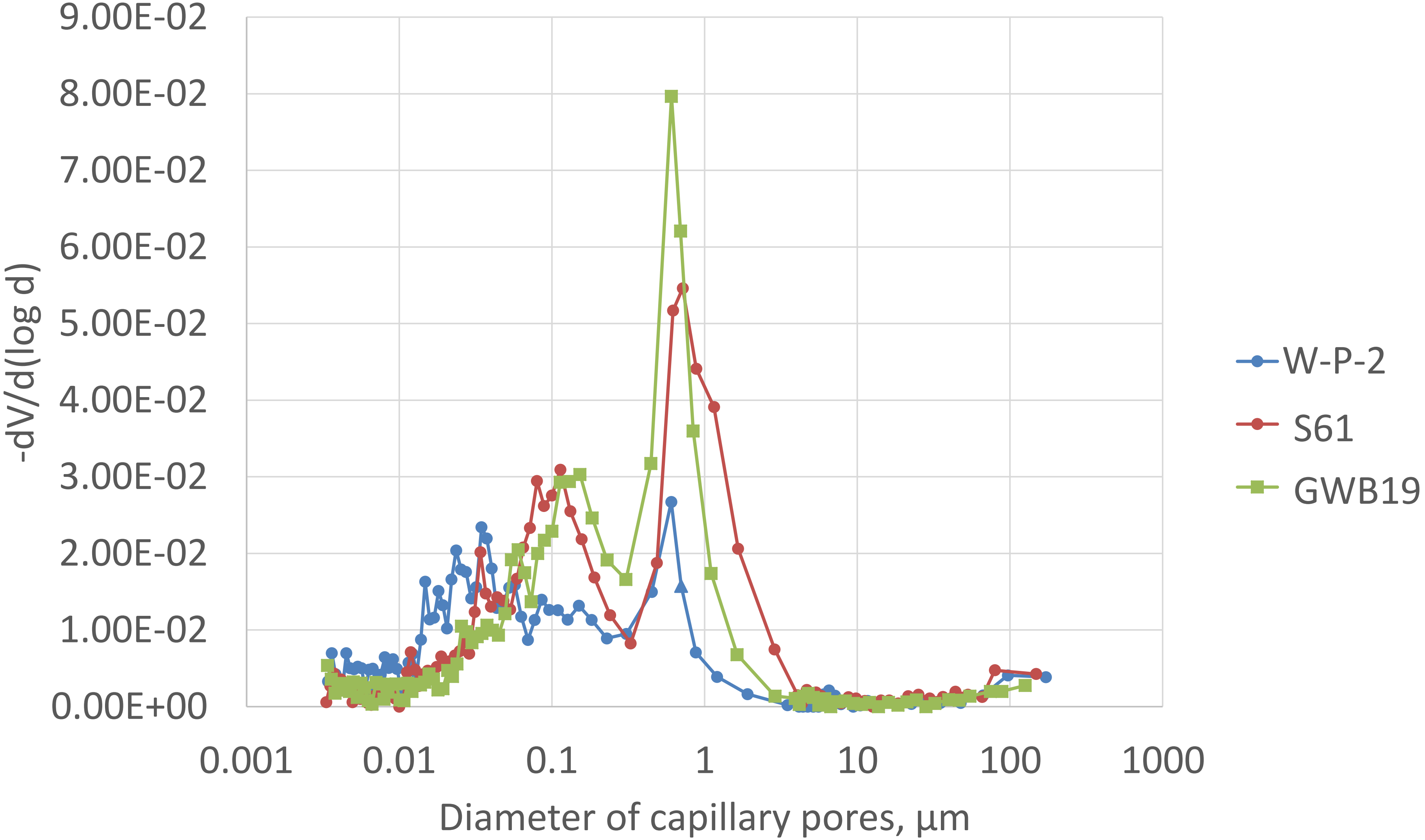
Auto Split: You can also use the Auto Split command to instruct Image-Pro Plus to analyze all existing outlines and automatically split any clustered objects it finds. Of course, not all clustered objects can be separated with Auto Split; in general, circular objects with minimal overlap work best.

Results

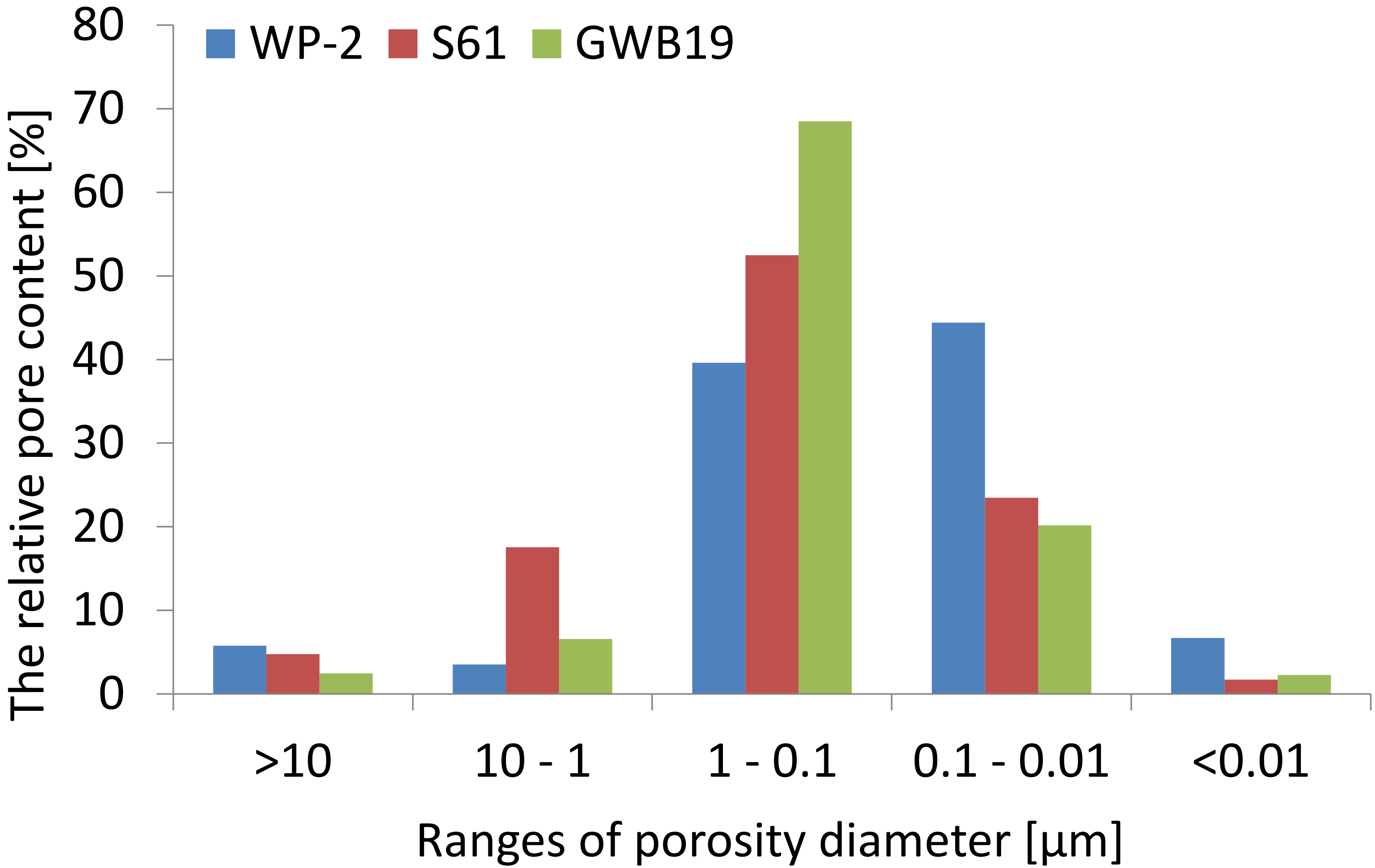
Mercury intrusion porosimetry



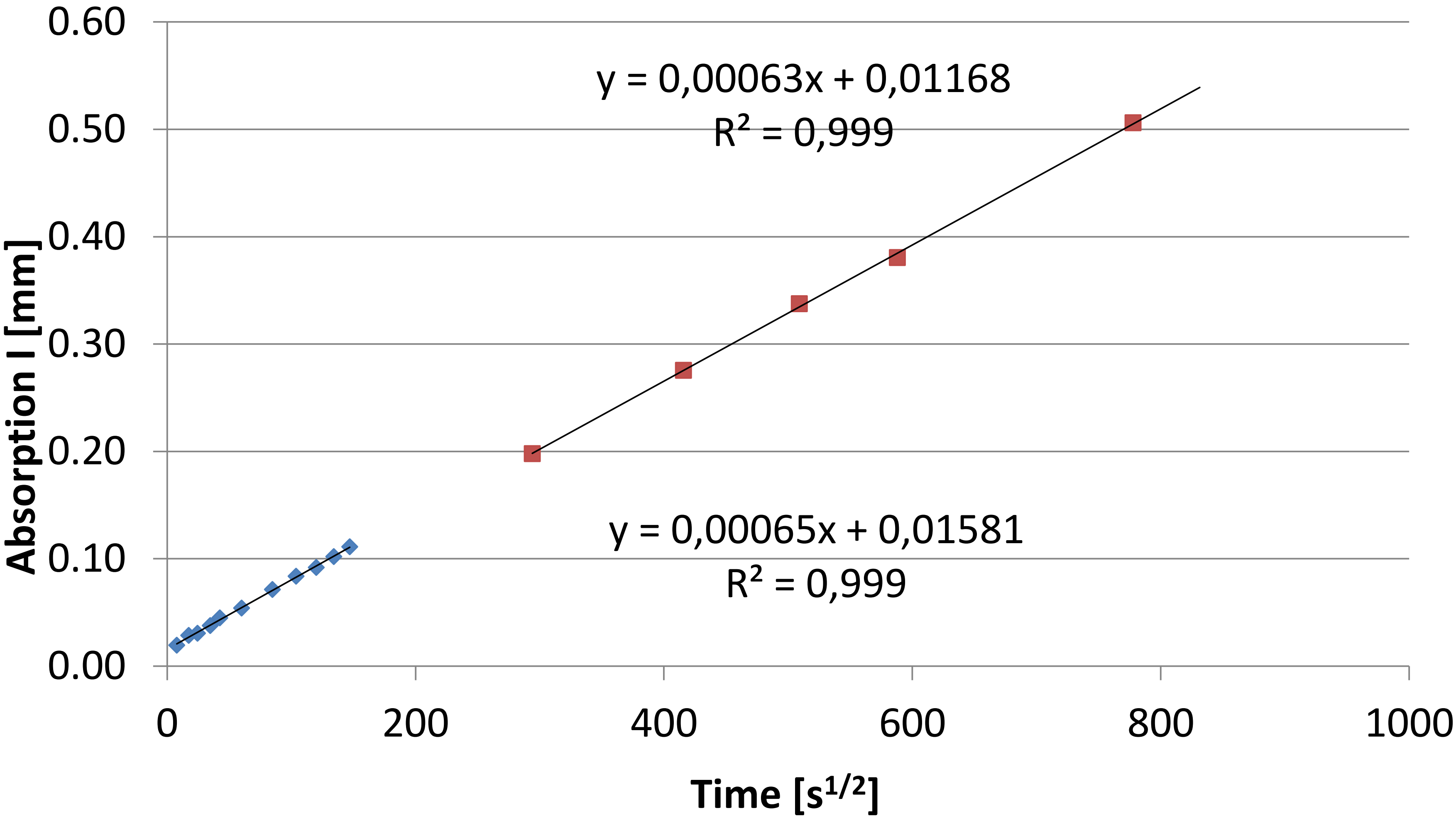
Mercury intrusion porosimetry



Mercury intrusion porosimetry

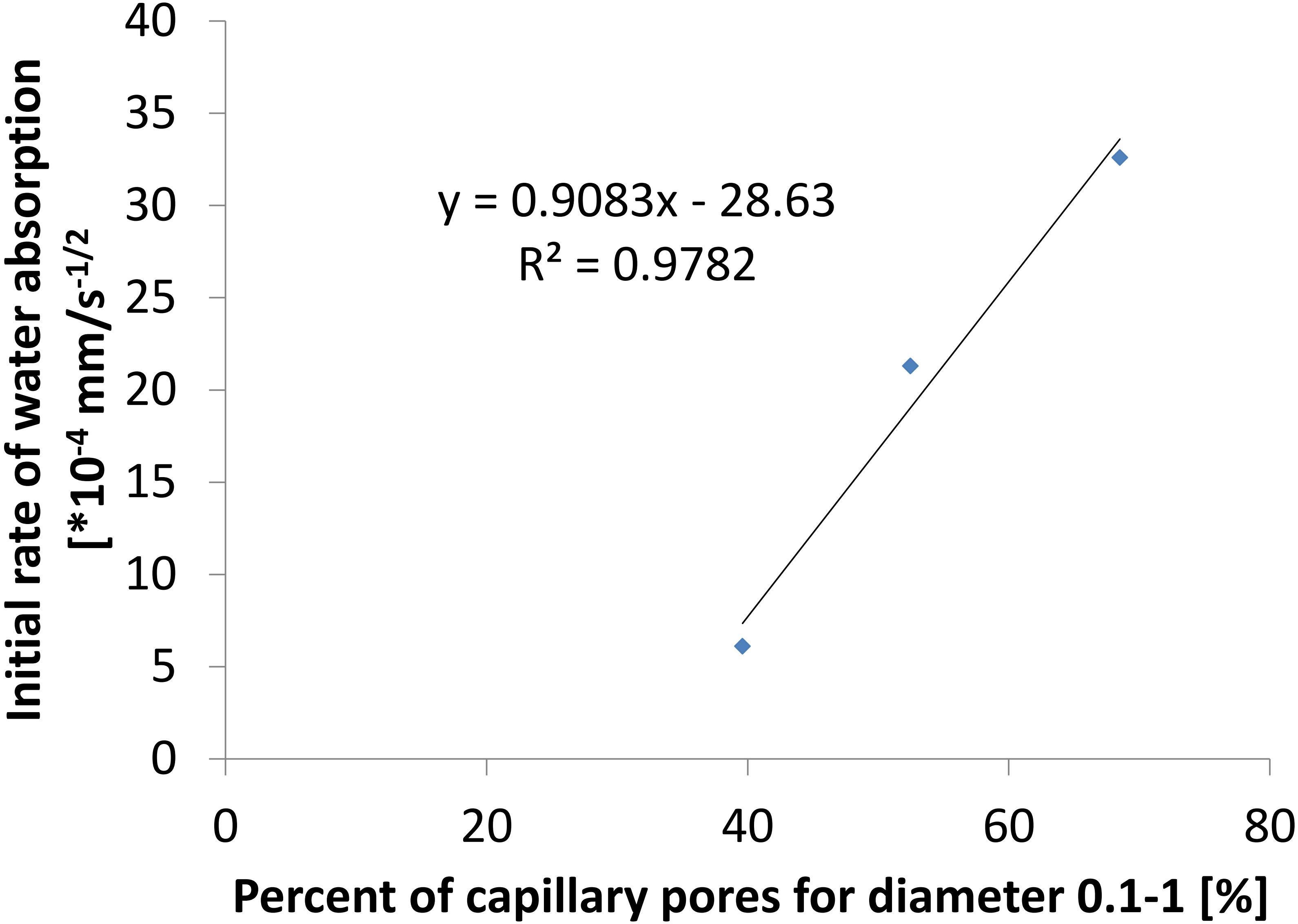


Water absorption



The example plot of the absorption *I* versus square root of time for W-P2 concrete

Capillary pores vs water absorption



EN 480-11 procedure

WP-2

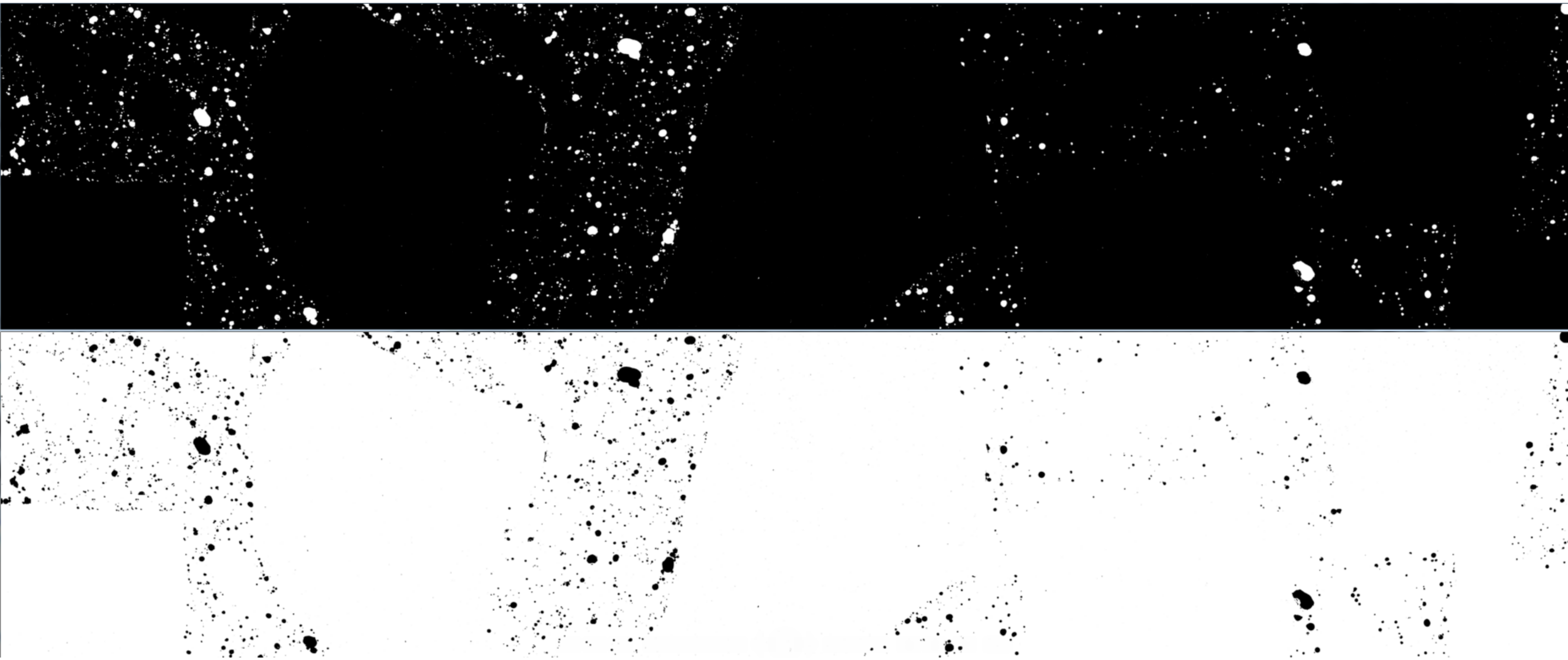
	Total length of the traverse line, Ttot=			1207,62	mm					
Column	1	2	3	4	5	6	7	8	9	10
	Air voids class	Class range	Numeber of chords in class	Frequency of chords	Share of counted air voids	Potential number of chords	Number of air voids in class	Singular air void volume	Air content	Cumulated air content
		µm		mm^{-1}	mm^2	mm^{-3}	mm^{-3}	mm^3	%	%
	1	0 do 10	2	0,001656151	0,0001178	14,0590094	-19,0760659	0,000000524	-0,0010	0,00
	2	15 do 20	11	0,009108832	0,0002749	33,1350752	25,46770809	0,000004190	0,0107	0,01
	3	25 do 30	4	0,003312303	0,000432	7,66736716	-21,8565526	0,000014100	-0,0308	-0,02
	4	35 do 40	21	0,017389589	0,000589	29,5239197	16,20545325	0,000033500	0,0543	0,03
	5	45 do 50	12	0,009936908	0,0007461	13,3184665	5,067048321	0,000065400	0,0331	0,07
	6	55 do 60	9	0,007452681	0,0009032	8,25141816	3,162278936	0,000113000	0,0357	0,10
	7	65 do 80	14	0,011593059	0,002278	5,08913922	0,814832683	0,000268000	0,0218	0,12
	8	85 do 100	15	0,012421135	0,002906	4,27430654	2,399771952	0,000524000	0,1257	0,25
	9	105 do 120	8	0,006624605	0,003534	1,87453459	-0,11459742	0,000905000	-0,0104	0,24
	10	125 do 140	10	0,008280757	0,004163	1,989132	0,606413318	0,001440000	0,0873	0,33
	11	145 do 160	8	0,006624605	0,004791	1,38271869	0,924289648	0,002140000	0,1978	0,52
	12	165 do 180	3	0,002484227	0,005419	0,45842904	0,321502712	0,003050000	0,0981	0,62
	13	185 do 200	1	0,000828076	0,0060476	0,13692633	-0,11114906	0,004190000	-0,0466	0,58
	14	205 do 220	2	0,001656151	0,006676	0,24807539	0,134702492	0,005580000	0,0752	0,65
	15	225 do 240	1	0,000828076	0,007304	0,1133729	-0,40854545	0,007240000	-0,2958	0,36
	16	245 do 260	5	0,004140378	0,007933	0,52191835	0,328465329	0,009200000	0,3022	0,66
	17	265 do 280	2	0,001656151	0,008561	0,19345302	-0,16701086	0,011500000	-0,1921	0,47
	18	285 do 300	4	0,003312303	0,009189	0,36046388	0,231680732	0,014100000	0,3267	0,79
	19	305 do 350	4	0,003312303	0,02572	0,12878315	0,044998092	0,022400000	0,1008	0,89
	20	355 do 400	3	0,002484227	0,02965	0,08378506	0,05912527	0,033500000	0,1981	1,09
	21	405 do 450	1	0,000828076	0,03358	0,02465979	0,00257777	0,047700000	0,0123	1,10
	22	455 do 500	1	0,000828076	0,0375	0,02208202	0,013675158	0,065400000	0,0894	1,19
	23	505 do 1000	6	0,004968454	0,591	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	24	1005 do 1500	0	0	0,9837					
	25	1505 do 2000	0	0	1,376					
	26	2005 do 2500	0	0	1,769					
	27	2505 do 3000	0	0	2,162					
	28	3005 do 4000	0	0	5,502					

Zawartosc zaczynu cementowego w betonie P=	26,3	%
Calkowita dlugosc cieciw przypadajaca na pory T_a=	19,323	mm
Calkowita zawartosc powietrza A=	1,60	%
Calkowita liczba mierzonych cieciw N=	147	
Powierzchnia wlasciwa porow α=	**30,43**	mm^{-1}
Stosunek zaczyn/powietrze R=	16,437	
Wskaznik rozmieszczenia L=	**0,26**	mm
Zawartosc mikroporow A300=	0,79	%

EN 480-11 procedure

Air void characteristics	Concrete type		
	S61	W-P-2	GWB19
A [%]	6.56	1.60	4.18
α [mm^{-1}]	20.26	30.43	33.29
L [mm]	0.19	0.26	0.17
A_{300} [%]	1.89	0.79	2.28

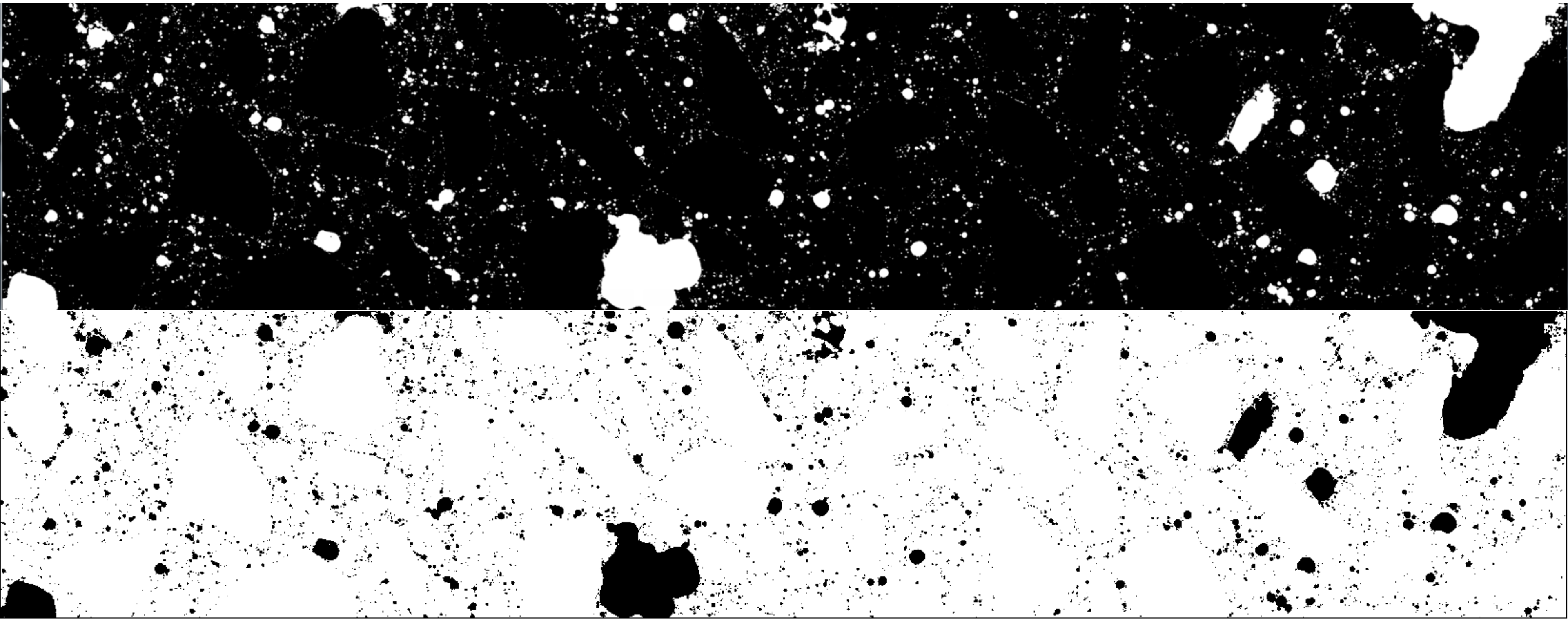
Image binarization



WP-2-1

15.83 x 76.48 mm

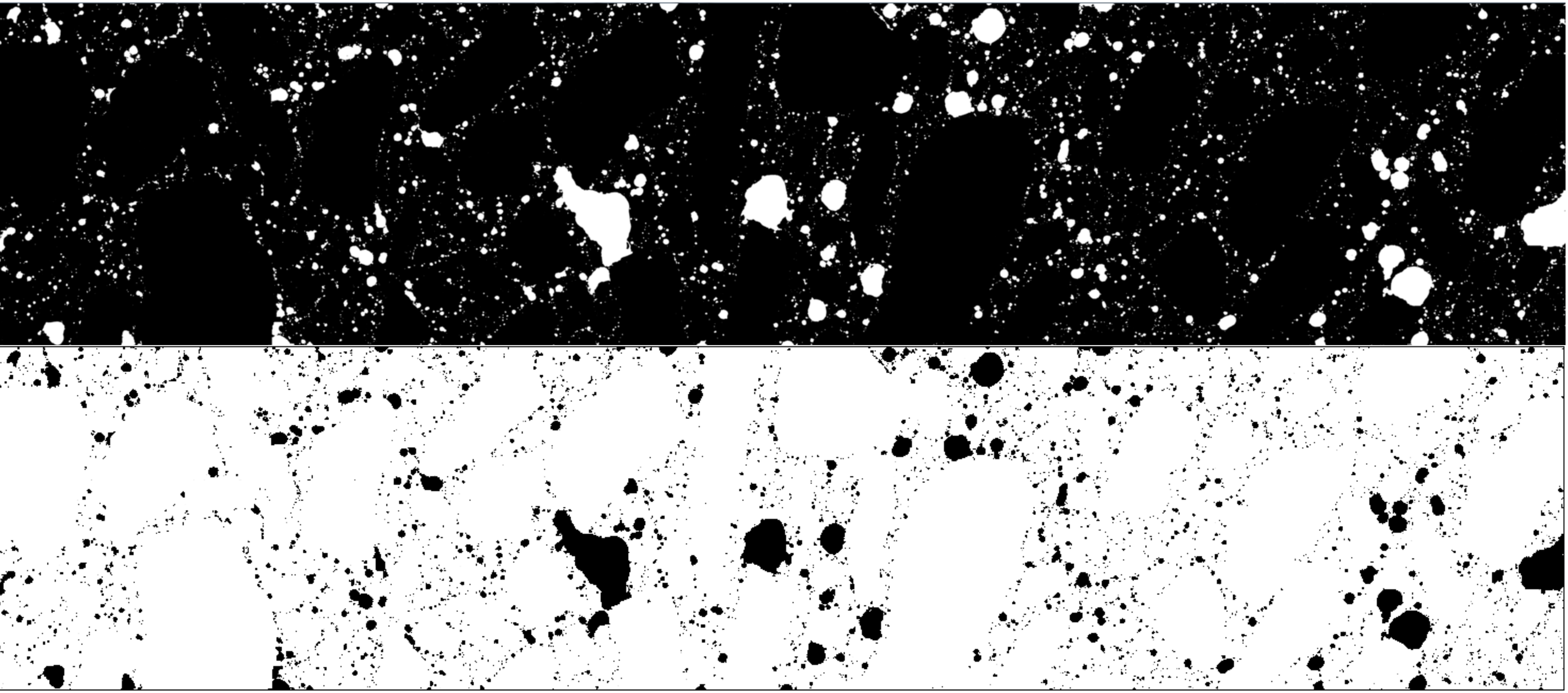
Image binarization



GWB19-1

15.83 x 76.48 mm

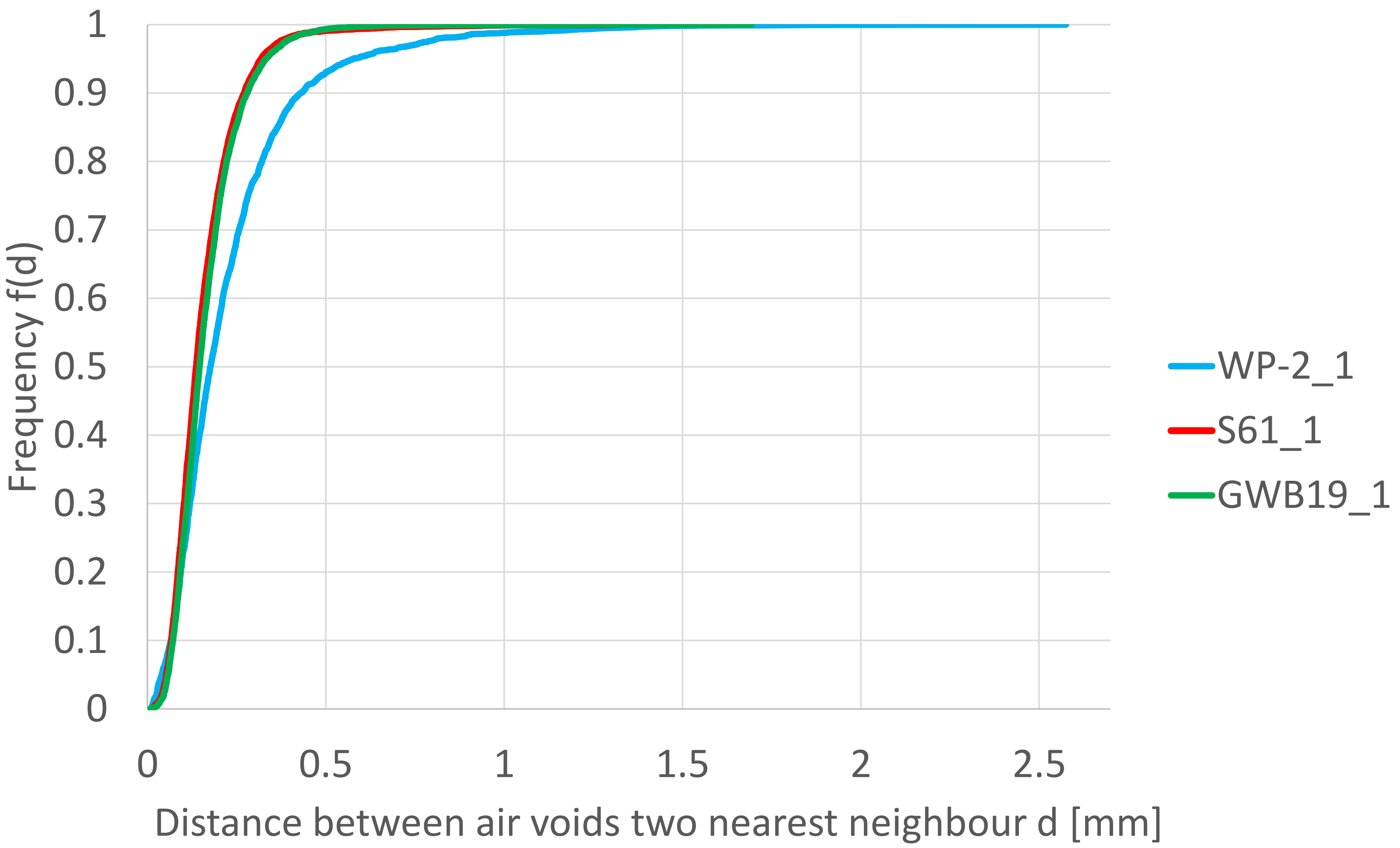
Image binarization



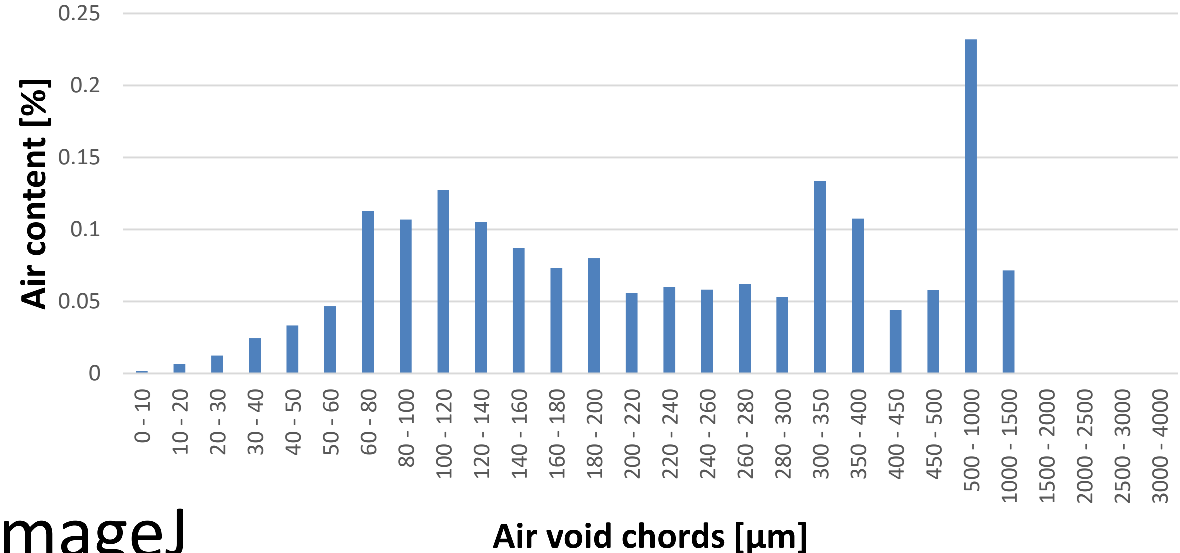
S61-1

15.83 x 76.48 mm

Air voids distribution



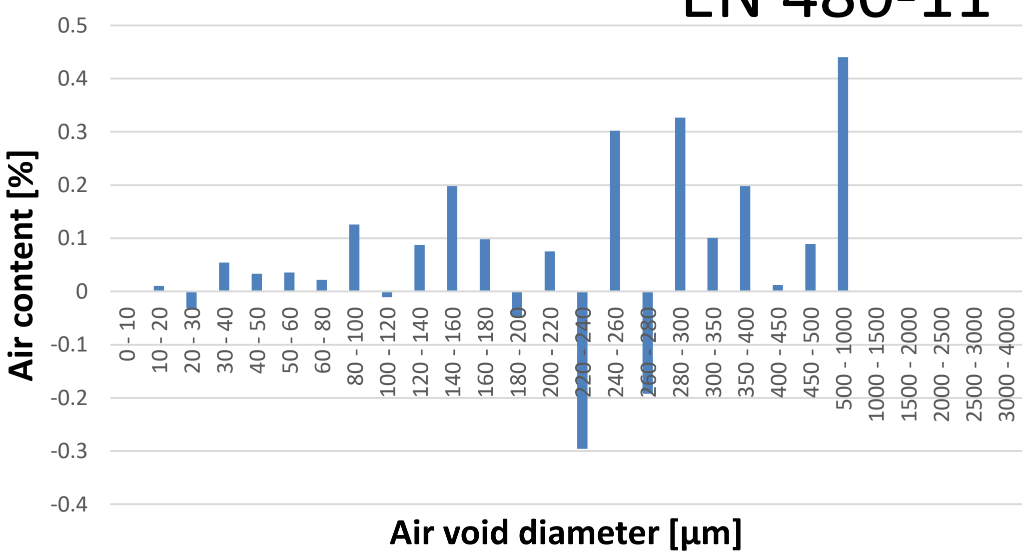
Air voids distribution



WP2

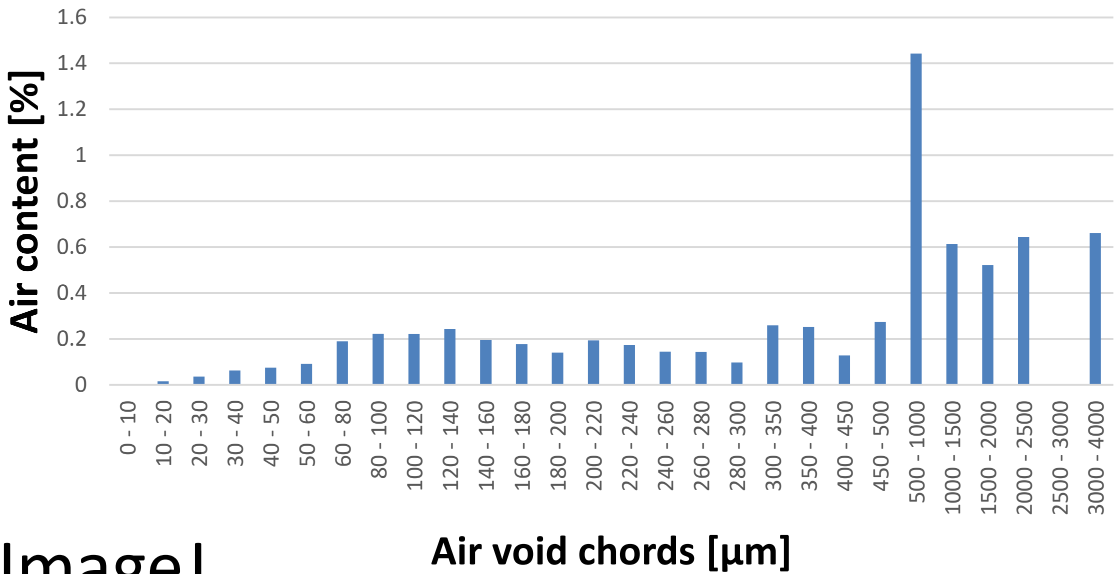
ImageJ

EN 480-11



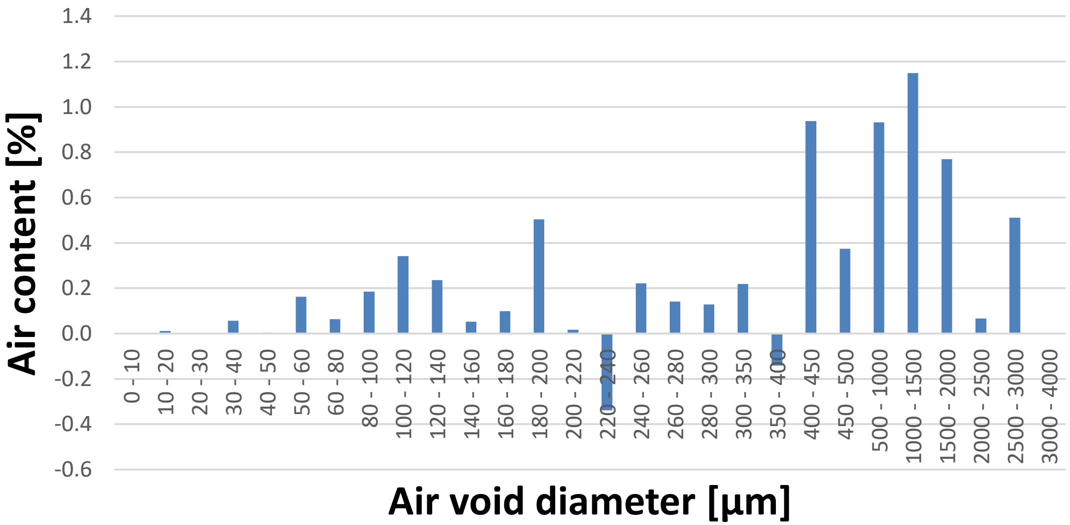
Air voids distribution

S61

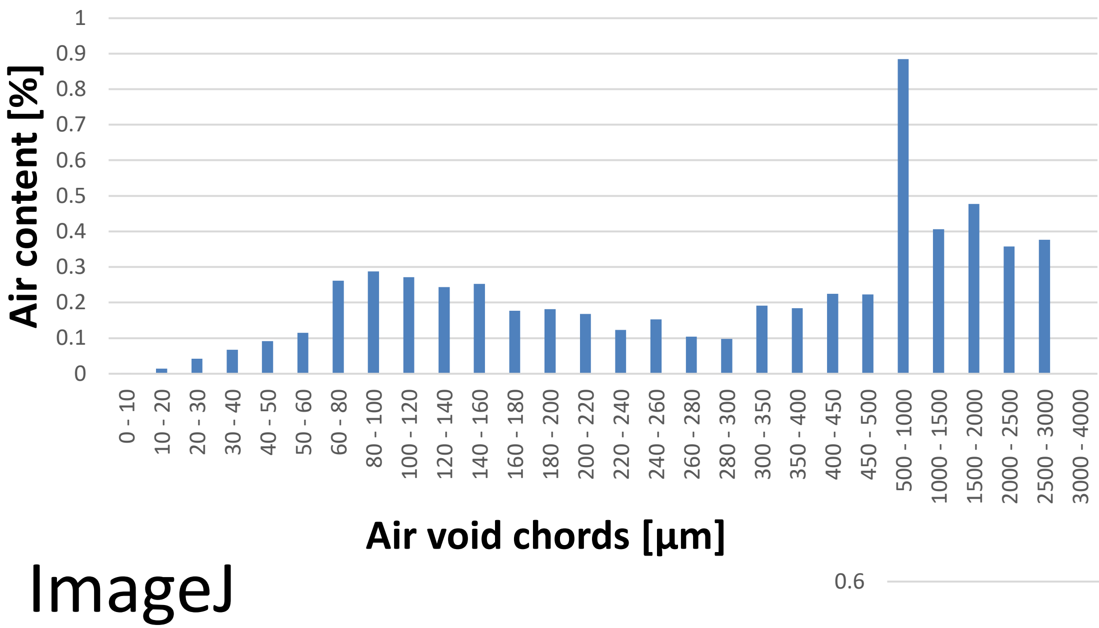


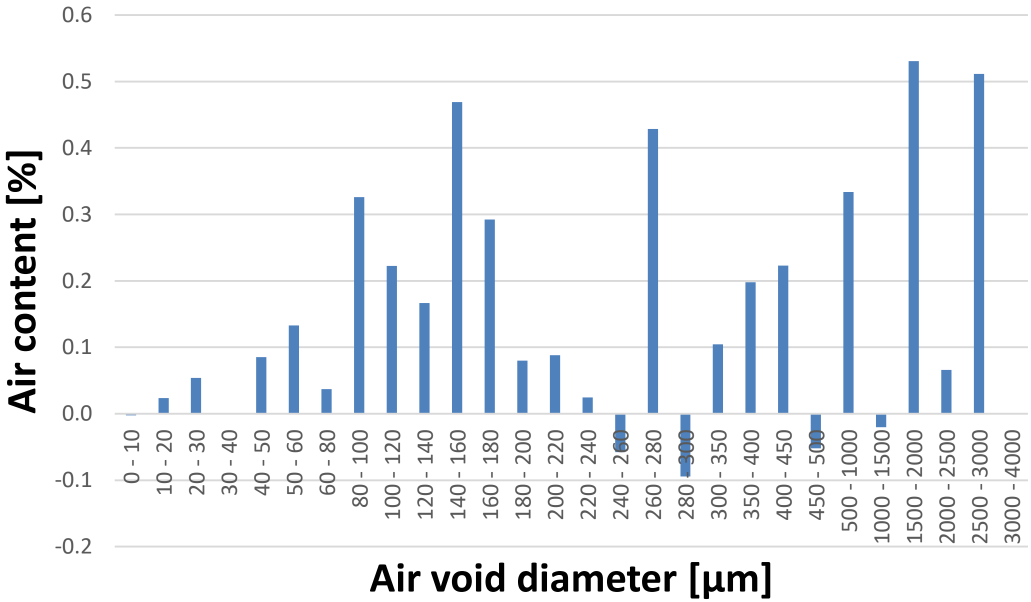
ImageJ

EN 480-11



Air voids distribution





Air voids distribution (image analysis)

	WP-2_1		S61_1		GWB19-1	
	ImageJ	Image Pro	ImageJ	Image Pro	ImageJ	Image Pro
Total air content [%]	1.75	1.70	7.65	6.95	9.01	7.03
Average distance between air void nearest neighbour [mm]	0.229	0.219	0.158	0.129	0.164	0.144
Average distance between center (x, y) of air void nearest neighbour [mm]	-	0.324	-	0.191		0.216
Average minor diameter of air void [mm]	0.053	0.048	0.058	0.052	0.059	0.054
Average major diameter of air void [mm]	0.069	0.069	0.081	0.082	0.079	0.081
Average diameter of air void [mm]	0.061	0.058	0.069	0.064	0.069	0.065
A_{300} [%]	1.12	1.10	2.47	2.53	2.70	2.55
Numer of air voids	3123	3123	6644	6590	7250	7273

Air voids distribution (image analysis)

Air void characteristics	S61_1			WP-2_1			GWB19_1		
	Traverse method	ImageJ	Image Pro	Traverse method	ImageJ	Image Pro	Traverse method	ImageJ	Image Pro
A [%]	6.56	7.65	6.95	1.60	1.75	1.70	4.18	9.01	7.03
L [mm]	0.19	0.16	0.13	0.26	0.23	0.22	0.17	0.16	0.22
A_{300} [%]	1.89	2.47	2.53	0.79	1.12	1.10	2.28	2.70	2.55

Further work (problems to be solved)

- Image processing – morphological filters
- Merging of air voids – problems with automatic spliting
- Setting shape parameters for automatic removal of cracks, non-air voids objects
- Comparing 2D-image with 3D-techniques – distances between centroids (x, y) instead of distances between air voids…?

Conclusions

- Quite good agreement of results obtained by linear traverse method (EN 480-11) and surface analysis using image analysis was observed:
  - Total air content
  - Content of micropores
  - Avarage distance between air voids (L) (void separation)
- 2D image analysis allows for a more accurate results of air voids distribution and evaluation of homogenity
- Image processing requires more detailed study
- Comparing the distance between pore centers may be more suitable for further comparison with 3D techniques

Thank you for your attention!

The research was founded by Polish National Centre for Research and Development (Project V4-Korea/2/2018, RADCON)