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Geometrical roughness analysis of cement paste surfaces using Coherence Scanning Interferometry and Confocal Microscopy

K.L. Apedo ^a, P. Montgomery ^b, N. Serres ^c, C. Fond ^a, F. Feugeas ^c

^a ICube, UMR CNRS 7357, Université de Strasbourg, 72 Route du Rhin, F-67400, Illkirch, France;

^b ICube, UMR CNRS 7357, Université de Strasbourg, 23 rue du Loess, 67037, Strasbourg, France

^c ICube, UMR CNRS 7357, INSA de Strasbourg, 24 Bld de la Victoire, 67084, Strasbourg, France;

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1. Introduction

Cement-based materials are heterogeneous, porous and rough composite materials with very complicated microstructures. During their service life, these materials are exposed to environments containing biological agents (microorganisms) and chemical compounds which may or may not be aggressive. The deterioration of cement-based material structures usually starts at the surface and progresses into the material [2]. The main factors that allow the penetration of aggressive agents into cement-based materials are their porosity [3–5] and their roughness [3, 4, 6, 7], both of which influence their bioreceptivity which is the ability of the material to be colonized by one or more groups of living organisms [3, 8]. Among the microorganisms able to colonize surfaces, bacteria are known to participate in the first step of biofilm formation.

The size of a bacterium ranges from 0.1 μm to 10 μm and its shape is variable, ranging from a sphere (for cocci) to rod-shaped (for bacilli) and spiral (for vibrios). Bacterial colonies form clusters which have a size of several tens of micrometers. The multiscale characterization of material surfaces appears to be an important area of the investigation to help provide a better understanding of how these external agents can form biofilms and interact with these surfaces.

The factor that is studied in this paper is the surface roughness. To characterize cementitious material surfaces, standard roughness parameters are often used with topographic reconstruction techniques such as confocal microscopy [1, 9–25] and atomic force microscopy (AFM) [26–41].

Using scanning electron microscopy (SEM) or SEM coupled with energy-dispersive X-ray (EDX), some studies allow the detection of the formation of the microstructures and their chemical composition or provide qualitative analysis of hydration processes and products of cement-based materials [27–32, 41–45].

More recently, [1] have introduced a multiscale analysis of cement paste surface roughness. Two new optical profilometry techniques, coherence scanning interferometry (CSI) and scanning confocal microscopy (SCM) have been used in the surface reconstruction. A new method named “window resizing” has been introduced in the calculation of the standard roughness parameters. The information about the characteristics of the techniques already used in cement-based surface analysis have also been reported in this paper.

26 The knowledge of the standard roughness parameters provides the necessary information to understand
27 the process of surfaces colonization by the microorganisms. But this information becomes very limited if it is
28 necessary to go deeper into the knowledge concerning the relation linking the size of the microorganisms and
29 the roughness geometry.

30 At this point, it then becomes interesting to determine the geometric roughness parameters. This consists
31 of knowing the developed lengths and surfaces.

32 It should be noted, however that these geometrical parameters may depend on the measurement technique
33 (in terms of its resolution and accuracy). It is also important to consider that the developed surface is a
34 parameter that has no meaning in itself. Only the surface viewed by a probe of a given size has a meaning.
35 Indeed, when the accuracy and the resolution increase (the probe size decreases for example), the surface viewed
36 by the probe increases until it reaches a certain limit. The notion of surface appears subjected to different
37 interpretations with no precise signification and only its projection is quantifiable.

38 Today, although many studies of cement-based materials have been performed using statistical roughness
39 parameters, to our knowledge, very few investigations have been addressed using geometrical roughness param-
40 eters. These parameters, known as roughness RN numbers make it possible to better quantify the developed
41 surface area available for colonization or reaction.

42 The studies already performed with roughness RN numbers deal with fracture surfaces of hydrated cement-
43 based materials using confocal microscopy [9–12, 16, 25, 46, 47]. Using a magnification of $\times 90$, (which controls
44 the lateral resolution) and a z slice of $10 \mu m$ (which controls the z resolution), [9] compared the roughness
45 numbers of several specimens of hydrated cement pastes and mortars. A great deal of their study was devoted
46 to the implementation of the confocal technique and software processing of confocal optical sections in digital
47 surface topographic maps. It has also been found that the fracture surface areas of cement paste are 1.8 times
48 greater than the nominal projected surface areas and that of mortars range from 2.4-2.8 times greater than the
49 nominal surface areas.

50 [10] have extended the analysis of [9] to the study of the correlation between the roughness numbers of
51 fracture surfaces and the mechanical parameters such as critical stress intensity factor K_{Ic} , critical effective
52 crack length a_c , compressive strength σ_c , total porosity and effective pore diameter. These analyses pointed
53 to a strong correlation between roughness numbers and stress intensity factor K_{Ic} as well as crack length a_c
54 whereas only a weak correlation has been observed with the compressive strength σ_c . Almost no correlation
55 has been found for all the other material properties (total porosity and effective pore diameter). The paper
56 also addresses the fractal dimension calculation as a function of the roughness numbers. Testing a notched
57 concrete beam using three-point bending, [11] used confocal microscopy to analyze the region near the interface
58 between the cement paste and the aggregate. The roughness numbers in the proximity of the paste-aggregate
59 interface has been found to be higher than that of the paste outside the interface. A correlation between the
60 critical stress intensity factor K_{Ic} , the critical crack extension δa_c and the roughness of the fracture surfaces
61 of cement-based materials has been found. [46] analyzed using confocal microscopy, the relationship between a
62 cement based material's strength and its roughness RN number. Cement based matrices reinforced by randomly

63 dispersed microfibers have been tested using both uniaxial tensile and three-point bending. [12] obtained for
64 cement pastes, the relation $\frac{K_{Ic}}{K_{Im}} = \sqrt{RN}$ initially established by [47] for S_i single fractured crystals. [12]
65 also established the relation between the roughness number and the fracture toughness values for mortars as
66 $\frac{K_{Ic}}{K_{Im}} = RN^{0.45}$. [16] demonstrated the potential applications of confocal microscopy through surface roughness
67 measurements using RN numbers. [25] introduced a new roughness parameter known as fractal roughness
68 number Rn_o which is scale-dependent only within the region of fractality of fractured cement pastes. This new
69 parameter has been shown to be lower than the ordinary roughness number RN . The relationship between
70 the water-to-cement ratio w/c and both RN and Rn_o have been provided. The correlation between fractured
71 cement paste compressive strength and the roughness numbers RN and Rn_o has also been investigated.

72 All these studies lead us to conclude that the roughness RN numbers are very useful for the characterization
73 of cementitious materials. Knowing that these roughness parameters depend on the measuring scale, multiscale
74 analysis seems to be required.

75 Although CSI showed its performance in the characterization of various kinds of materials [48–58] its use
76 remains relatively unexplored in the field of cement-based materials [1, 59, 60].

77 In view of the these previous studies, therefore, two things become quite clear. Firstly, the quantitative
78 analysis of the surface roughness of cementitious materials using RN numbers is very important. Secondly,
79 CSI has a great potential in this area but requires careful study to explore the performance, limitations and
80 protocols for successful measurement in view of the high roughness and inhomogeneous nature of cementitious
81 materials.

82 The present paper extends the work presented in [1]. Both polished and unpolished cement paste surfaces
83 already measured with CSI and SCM and presented in [1] are used to quantify multiscale roughness RN numbers
84 introduced by “window resizing”. The paper describes the roughness RN numbers calculation method using
85 “window resizing”. The identification of fractal region and its fractal dimension of both polished and unpolished
86 cement pastes is also investigated.

87 2. Method

88 2.1. CSI and SCM

89 The microscopy techniques (CSI and SCM) used in this paper have already been described in [1]. CSI
90 and SCM are two optical profiling techniques for measuring a material surface’s topographic map. These two
91 optical techniques are different due to their accuracy in Z ($0.04 \mu m$ for CSI and $0.1 \mu m$ for SCM), their lateral
92 resolutions ($0.45 \mu m$ for CSI and $2 \mu m$ for SCM), their Z -resolutions ($1 nm$ for CSI and $10 nm$ for SCM) and
93 the extent of the surfaces that they allow to explore ($184 \mu m \times 138 \mu m$ for CSI and $4.5 mm \times 4.5 mm$ for
94 SCM). Thus, the two techniques make it possible to measure the topographic map of surfaces at two different
95 scales. Other characteristics of these techniques can be found in [1].

96 Areas selected in the middle of the samples were scanned by both techniques.

97 Using CSI with a camera pixel size of $0.13 \mu m \times 0.13 \mu m$, the scanned areas consisted of $183 \mu m \times 138 \mu m$
98 for the polished samples and $178 \mu m \times 99 \mu m$ and $69 \mu m \times 55 \mu m$ in the case of the unpolished samples.

99 In the case of SCM, the scanned areas consisted of $2\text{ mm} \times 2\text{ mm}$ squares and the pixels were recorded
100 every $4\text{ }\mu\text{m} \times 4\text{ }\mu\text{m}$.

101 2.2. Geometrical parameters measurement by window resizing

102 Among the methods that allow the quantification of the surface roughness of cement-based materials,
103 one of the most widely used is the statistical analysis based on the determination of standard roughness
104 parameters such as the amplitude parameters, the spacing parameters and the hybrid parameters [1, 19–
105 27, 29, 33, 35, 39, 41, 61]. Among these standard roughness parameters may be mentioned: the altitude
106 difference between the highest and lowest measured points H_{mm} [1], the average of the absolute irregularities
107 R_a [1, 19–25, 29, 35, 41, 61], the root mean square (rms) of the irregularities R_q [1, 19, 23–27, 33, 35, 39, 61],
108 the skewness R_{ks} and the kurtosis R_{ku} [23, 25]. Some of these standard roughness parameters have already
109 been used in conjunction with the window resizing method [1].

110 A parameter that is also commonly used to quantify the roughness of cementitious materials is the roughness
111 number RN . This parameter is generally used to quantify the roughness of fractured surfaces [12, 16, 25, 46]
112 and was also used to quantify the roughness of rubber toughened polymethyl-methacrylate fracture surfaces
113 [62, 63]. It is defined as the sum of the areas of triangulated surfaces (A_i) in relation to the area of the
114 corresponding nominal surface (A_{pi}) :

$$RN = \frac{\sum A_i}{\sum A_{pi}} \quad (1)$$

115 The reference (nominal) surface is not easy to define. In the literature, a vertical projection of the developed
116 surface onto the horizontal xy -plane is often considered as the nominal surface [12, 16, 25, 29, 46]. More recently,
117 [24] have introduced a new reference surface consisting of the Fourier surface.

118 In this paper, the developed lengths and surfaces are used to quantify the roughness of cement paste surfaces.
119 The window resizing method whose basis has already been presented in [1], is adopted. Roughness numbers
120 using the developed lengths and surfaces are a new aspect of a roughness quantification implemented in window
121 resizing method.

122 According to this method, for a given integer δ (see [1]), the developed lengths and surfaces are calculated
123 on each cell and then the sum of these lengths and surfaces over all the cells is obtained; a cell being defined
124 as a set of points forming a square (see Fig. 1).

125 Concerning the roughness parameters along x and y , the principle is to calculate the ratio between the
126 developed length and the reference length along x and y . Thus, a line is defined by specifying either j in the x
127 direction or i in the y direction. The reference (nominal) length (l_{0x} or l_{0y}) is calculated from the end points for
128 each line. The lengths of straight segments between these end points are added up and the sum is calculated
129 in relation to the reference length for each line. The average of the length report is then obtained for x and y
130 directions. The geometrical roughness numbers RN_x and RN_y are thus defined.

131 The developed surface is calculated in several ways. In the following, for a given δ , p_1 , p_2 , p_3 and p_4 denote
132 the vertices of a basic square and p_5 is the intersection of the two diagonals (Fig. 1).

133 The surface area (A_α) of each basic cell is calculated using different techniques :

- Surface from a diagonal, s_{14} and s_{23} : The diagonal p_1-p_4 is considered; then the surface s_{14} is calculated as the sum of the planar surfaces of the triangles $p_1-p_4-p_3$ and $p_1-p_4-p_2$. Considering the diagonal p_2-p_3 , the surface s_{23} is calculated as the sum of the planar surfaces of the triangles $p_2-p_3-p_1$ and $p_2-p_3-p_4$.
- Surface from a midpoint, s_{p_5} : The intersection of the two diagonals is defined by p_5 of which altitude is the average of those of p_1 , p_2 , p_3 and p_4 . s_{p_5} is then calculated as the sum of the planar surfaces of the four triangles thus defined.

All these surfaces are known as triangulated surfaces. Using “window resizing”, a new type of surface is introduced :

- Warped surface, s_{q4} : The altitude within a basic cell is interpolated using the interpolation functions of four-node quadrilaterals. The altitude is given by

$$\tilde{z} = \frac{1}{4}[z_{p_1}(1-\zeta)(1-\eta) + z_{p_2}(1-\zeta)(1+\eta) + z_{p_3}(1+\zeta)(1-\eta) + z_{p_4}(1+\zeta)(1+\eta)] \quad (2)$$

where z_{p_1} , z_{p_2} , z_{p_3} and z_{p_4} are the altitudes of the four vertices of the basic cell.

In the parametric space ζ and η range from -1 to +1. Using an isoparametric system, the coordinates (\tilde{x}, \tilde{y}) are interpolated in the same manner as the functions of (x_{p_1}, y_{p_1}) , (x_{p_2}, y_{p_2}) , (x_{p_3}, y_{p_3}) and (x_{p_4}, y_{p_4}) .

Thus, s_{q4} is given by

$$s_{q4} = \int ds = \int_{-1}^{+1} \int_{-1}^{+1} \|\vec{T}_\zeta \wedge \vec{T}_\eta\| \frac{\partial x}{\partial \zeta} d\zeta \frac{\partial y}{\partial \eta} d\eta \quad (3)$$

where \vec{T}_ζ and \vec{T}_η are the tangent vectors to the warped surface. These vectors are given by

$$\vec{T}_\zeta = \begin{bmatrix} \frac{\partial x}{\partial \zeta} \\ \frac{\partial y}{\partial \zeta} \\ \frac{\partial z}{\partial \zeta} \end{bmatrix} \quad \text{and} \quad \vec{T}_\eta = \begin{bmatrix} \frac{\partial x}{\partial \eta} \\ \frac{\partial y}{\partial \eta} \\ \frac{\partial z}{\partial \eta} \end{bmatrix} \quad (4)$$

The surface s_{q4} is then calculated using a numerical integration scheme.

We also introduce a new reference (nominal) surface :

- Reference surface : For a given δ and for each basic cell, the reference surface area (denoted by $A_{0\alpha}$) is the local plane of the least squares of the analyzed unit cell.

The ratio between the sum of the developed surface areas and the sum of the reference surface areas is then calculated as another roughness parameter (RN_S).

$$RN_S = \frac{A}{A_0}, \quad \text{with} \quad A = \sum_{\alpha=1}^N A_\alpha \quad \text{and} \quad A_0 = \sum_{\alpha=1}^N A_{0\alpha} \quad (5)$$

where N stands for the number of basic cells for a given δ .

In this paper, these new geometrical roughness parameters (RN_S , RN_x , RN_y) are used to compare the different surfaces studied by both techniques.

158 It is also important to note that the geometrical parameters may be from the sampling or from the convo-
159 lution.

160 For the sampling, the altitudes of the vertices are experimental values obtained from the optical techniques.
161 As the surface obtained by sampling is highly dependent on the measurement technique, (notably its accuracy
162 and resolution), in this paper, another kind of surface known as the convolution surface, is introduced. This
163 surface is one that will be viewed by the bacterial colonies of a given size. So, for the convolution, the altitudes
164 of the vertices are those of the center of a sphere; the radius of this sphere being $\delta dx - \epsilon$, with ϵ being a small
165 quantity compared with dx . Whatever the method (sampling or convolution), the points are taken every δ .

166 3. Materials

167 The materials used in this paper were already described in [1]. The cement pastes were manufactured using
168 the commercial anhydrous CEM I-52,5R cement (see [1] for its chemical composition) provided by HOLCIM
169 France. A water-to-cement ratio of $w/c = 0.4$ was used to guarantee optimum mechanical properties and the
170 samples were manufactured using the ASTM C191 and in accordance with the manufacturer's recommendations
171 for the water-to-cement ratio. Polished and unpolished disk-shaped samples with a diameter of 22 mm and a
172 thickness of 4 mm were measured using CSI and SCM (see [1] for the conservation of the samples, the curing
173 times and the polishing process). The polished samples were analyzed in order to go deeper in the understanding
174 of relations between cement surfaces parameters and biocolonisation as the factor that is studied in this paper
175 is the surface roughness. In the scale of investigation with CSI and SCM, the roughness numbers depend on the
176 bulk porosity of the samples. But the roughness of concrete is depending on its composition and its interactions
177 with the mould. So the polishing will allow us to erase the preparation effect and to highlight the impact of
178 porosity on roughness parameters. Note that the polishing was been made after 28 days of curing time, so,
179 those samples made of CEM I-52,5R are hard enough to be polished without damage. To polish samples of
180 this thickness, negatives dedicated to polishing were carried out. These are thicker than the negative used
181 for the implementation of the samples allowing them to exceed 1.5 mm and being polished with a secure and
182 repeatable way (see Fig. 2).

183 4. Results and discussion

184 The results of 3D topographic measurements of both polished and unpolished cement paste samples can be
185 found in [1].

186 4.1. Results on polished samples

187 After comparison, the roughness numbers RN_S obtained using the different calculation methods (s_{14} , s_{23} ,
188 s_{p5} and s_{q4} methods) lead to quite similar results. Thus, for the sake of clarity, in the following, only the results
189 provided by the warped surface method is presented.

190 Fig. 3 shows the evolution of the roughness number RN_S (the sampling is represented by the subscript e and
191 the convolution by the subscript c) as a function of δ (or the probe radius, R_p in the case of the convolution).
192 Fig. 3(a) corresponds to the evolution of the roughness number RN_S as a function of δ in the case of the samples

193 measured with the SCM. This roughness number decreases from 1.02 to 1 for the sample 1 and from 1.016 to 1
 194 for the samples 2 and 3. These results demonstrate the reproducibility of the samples. The value of $RN_S = 1$
 195 represents an asymptote to the plot in each case since a developed surface is always greater than a reference
 196 surface. A negligible discrepancy was obtained between the results of the three samples. Also, no major
 197 difference can be noted between sampling and convolution even if the surface viewed by the bacterial colonies
 198 (convolution) is always slightly smaller than the surface provided by the measurement technique (sampling).

199 Fig. 3(b) corresponds to the evolution of the roughness number RN_S as a function of δ in the case of the
 200 sample measured with CSI. This roughness number decreases from 1.79 to 1. Sampling and convolution plots
 201 overlap correctly for $\delta dx \leq 1 \mu m$. A bifurcation occurs for $\delta dx > 1 \mu m$. This information suggests that the
 202 value of $\delta dx = 1 \mu m$ is a critical value from which the developed surface obtained by the sampling is always
 203 very much greater than the surface resulting from the convolution which is the surface viewed by the bacterial
 204 colonies. For a bacterial size $< 1 \mu m$, the surface viewed by these bacteria is the surface obtained by CSI.
 205 Thus, the SCM technique does not make it possible to know the extent of the bacterial colonization. However
 206 for a bacterial size $> 1 \mu m$ the surface viewed by the bacteria is that obtained by the convolution with a sphere
 207 of the corresponding size.

208 Indeed, when $\delta > 8$, the discrepancy between the sampling and the convolution becomes large enough
 209 because many parts of the sampled surface become inaccessible to the convolution sphere. This effect is
 210 illustrated in Fig. 4. Thus, the surface viewed by the bacteria (convolution) is substantially smaller than the
 211 surface viewed by the measuring tool (sampling).

212 In addition, this critical value ($\delta dx = 1 \mu m$) approximately delimits the fractal region of the materials since
 213 for $\delta dx \leq 1 \mu m$, it is possible to find a linear region on the plot (from the second point of the plot to the
 214 bifurcation point). It is important to remember that, when the RN_S number is plotted as a function of δdx
 215 on a log-log system, the linear part of the plot determines the region of fractality and the slope of this part
 216 determines the fractal dimension as [25] : $D_S = 2 - \alpha$. In this case, after calculation, $D_S = 2.229$. Thus, only
 217 CSI allows access to the fractality region of cement pastes used in this study.

218 Fig. 3(c) is a superposition of the results from both measurement techniques. This figure shows that the
 219 roughness numbers obtained by SCM are always lower than those obtained by CSI. The results also show that
 220 CSI and SCM are two different techniques that allow the measurement of the roughness number of cement
 221 pastes at two different scales (two different resolutions and accuracies), as the SCM plots overlap with the plots
 222 of the results from CSI.

223 Fig. 5, 6, 7 and 8 show the evolution of the roughness numbers RN_x and RN_y as a function of δ (or the
 224 probe radius, R_p). The roughness numbers were measured along two perpendicular directions named x and y .

225 Figs 5(a) and 6(a) correspond to the evolution of the roughness numbers RN_x and RN_y as a function
 226 of δ for the case of the samples measured with SCM. For all these samples, the roughness numbers decrease
 227 approximately from 1.013 to 1. No significant discrepancies were obtained between the convolution and the
 228 sampling, even if for a given δ , the sampling results are always slightly greater than the convolution results.

229 Figs 5(b) and 6(b) correspond to the evolution of the roughness numbers RN_x and RN_y as a function of δ

230 in the case of the sample measured with CSI. These roughness numbers decrease from 1.457 to 1. Sampling and
 231 convolution plots overlap correctly for $\delta dx \leq 1 \mu m$. A bifurcation occurs for $\delta dx > 1 \mu m$. This information
 232 indicates once again that the value of $\delta dx = 1 \mu m$ is a critical value from which the roughness number
 233 obtained by the sampling is always greater than the roughness number resulting from the convolution. The
 234 same conclusion about the fractality region can also be drawn. Using the formula that provides the fractal
 235 dimension of profiles ($D_p = 1 - \alpha$, where α is the slope of the linear part of the plots in Figs 5(b) and 6(b)),
 236 respectively 1.140 and 1.143 were found as fractal dimensions for x and y directions. It can be noticed that
 237 $D_S \approx D_{px} + D_{py}$, where D_{px} and D_{py} are the fractal dimensions for x and y directions, respectively.

238 Figs 5(c) and 6(c) are the superpositions of the results from both measurement techniques. These figures
 239 show that the roughness numbers obtained with SCM are always lower than the sampling results obtained with
 240 CSI. The results show again, that CSI and SCM are two different techniques that allow the measurement of
 241 the developed length of cement pastes at two different scales, as the SCM plots overlap with the plots of the
 242 results from CSI.

243 It is important to note that whatever the technique used, the roughness numbers measured along the two
 244 perpendicular directions (x and y), give identical results (see figs 7 and 8). These results might indicate an
 245 isotropy of these polished surfaces in terms of their roughness. The values obtained for fractal dimensions for
 246 both directions also support this conclusion.

247 In the literature, while several authors have addressed the problem of the roughness of cementitious polished
 248 samples using standard roughness parameters [26, 27, 29–33, 40, 41], to our knowledge, no roughness studies
 249 have been performed using RN numbers. Also, except [1] who have adopted a comparative study between SCM
 250 and CSI, all existing results use the AFM technique which has major drawbacks of a very small size of the area
 251 of interest ($150 \times 150 \mu m$ and a maximum height of a few μm) and a long scanning time [1, 53].

252 4.2. Results on unpolished samples

253 As in the case of the polished samples, the surfaces obtained using the different calculation methods lead
 254 to quite similar results. Only the results provided by the warped surface technique is then presented in the
 255 following.

256 The evolution of the roughness number RN_S as a function of δ (or the probe radius, R_p) is presented in
 257 Fig. 9. In the case of the samples analyzed with SCM (Fig. 9(a)), this number decreases from 1.250 to 1.002
 258 for the sample 1, from 1.232 to 1 for the sample 2 and from 1.222 to 1 for the sample 3. The plots of the
 259 convolution and the sampling overlap each other fairly well and the sampling results are slightly greater than
 260 the convolution results.

261 Fig. 9(b) corresponds to the evolution of the roughness number RN_S as a function of δ in the case of
 262 the sample tested with CSI. The ratio decreases from 1.988 to 1.041 for sample 1 and from 1.941 to 1.025 for
 263 sample 2. The sampling and the convolution plots overlap correctly for $\delta dx \leq 1 \mu m$. A bifurcation occurs for
 264 $\delta dx > 1 \mu m$, leading to the same conclusions as in the case of the polished samples.

265 The fractal region of these unpolished samples is once again accessible only with CSI. This region corresponds
 266 to the points on the plot from the third point on the plot to a point near to the bifurcation point. The calculation

267 gives $D_S = 2.175$ and $D_S = 2.157$ respectively for samples 1 and 2. These fractal dimensions are comparable
268 with the value reported in [25] ($D = 2.305$), and in [64] (Fractal dimension ranging from 2.02 to 2.12). The
269 first authors have tested fractured cement pastes with $w/c = 0.4$ after 28 days of hydration and the second
270 ones have studied fractured cement pastes with $w/c = 0.3$ and 0.5 after 23 days of hydration.

271 For the profile fractal dimensions, 1.107 was found as a fractal dimension for both the x and y directions in
272 the case of sample 1 and 1.095 and 1.100 for respectively the x and y directions in the case of sample 2.

273 Fig. 9(c) is a superposition of the results from both measurement techniques and serves as a tool for
274 comparing the results from each technique.

275 The evolution of the roughness numbers RN_x and RN_y as a function of δ are presented in Figs 10, 11, 12
276 and 13.

277 Figs 10(a) and 11(a) correspond to the evolution of the roughness numbers RN_x and RN_y as a function of
278 δ for the case of the samples tested with SCM. For all these samples, the ratios decrease approximatively from
279 1.013 to 1. No significant discrepancies were obtained between the convolution and the sampling.

280 Fig. 10(b) and 11(b) correspond to the evolution of the roughness numbers RN_x and RN_y as a function
281 of δ in the case of the samples tested with CSI. These numbers decrease from 1.584 to 1.048 for sample 1 and
282 from 1.551 to 1.025 for sample 2. The sampling and the convolution plots overlap correctly for $\delta dx \leq 1 \mu m$
283 and a bifurcation occurs for the same value of δ as in the previous cases.

284 Figs 10(c) and 11(c) are the superpositions of the results from both measurement techniques. These figures
285 show that the roughness numbers obtained with SCM are always lower than the sampling results obtained with
286 CSI.

287 It is also important to note that as in the case of the polished samples, whatever the technique used, the
288 roughness numbers measured along the two perpendicular directions (x and y), give identical results (see figs
289 12 and 13) indicating an isotropic character of these unpolished cement paste surfaces.

290 Using the results of this study, CSI appears as a very interesting tool for studying both polished and
291 unpolished cement paste surfaces. It allows the overcoming of the problems of measurement of too smooth
292 cementitious surfaces with a confocal microscope or very rough cementitious surfaces with AFM.

293 5. Conclusions

294 In this paper, roughness numbers of both polished and unpolished cement paste samples have been analyzed.
295 The surface of these heterogeneous and porous materials have been characterized using CSI and SCM. The
296 data from both techniques have been used by “window resizing” in the calculation of the roughness numbers
297 along profiles (RN_x and RN_y) and on an entire surface (RN_S). The roughness numbers of Both polished and
298 unpolished samples have been quantified. These roughness parameters can be used as quantification tools of
299 the bacterial colonization extent at a given scale.

300 Despite the limitations of these measuring techniques, the study clearly shows the ability of CSI to analyze
301 very porous and rough surfaces such as cement pastes. The results show that CSI and SCM are two tools that
302 allow the measurement of the surface roughness of the cement pastes at two different scales. The “window

303 resizing” technique allows the use of two methods of the calculation of the roughness numbers : the sampling
304 and the convolution. The sampling is the parameter related to the measuring tool while the convolution is the
305 parameter related to the roughness viewed by a bacterium of a given size.

306 The quantification of developed lengths and surfaces will be very useful in the further analysis of the
307 colonization of cement-based material surfaces by microorganisms and also for other subsequent studies. Four
308 calculation methods of developed surfaces have been implemented in “window resizing” and the results from
309 these methods are identical. The results also show that in term of roughness, cement pastes are relatively
310 isotropic, whether polished or not.

311 CSI makes it possible to quantify more precisely the surface available for the colonization of bacteria than
312 SCM.

313 In the case of SCM, whether the samples are polished or not, no major difference can be remarked between
314 sampling and convolution even if the surface viewed by the bacterial colonies (convolution) is always slightly
315 smaller than the surface provided by the measurement technique (sampling).

316 Using CSI, whether the samples are polished or not, sampling and convolution plots overlay correctly for
317 $\delta dx \leq 1 \mu m$. A bifurcation occurs for $\delta dx > 1 \mu m$ and the surface viewed by the bacteria (convolution) is
318 substantially smaller than the surface viewed by the measuring tool (sampling).

319 It has been found that only CSI allows access to the fractality region of cement pastes used in this study.

320 In future work, the CSI and “window resizing” techniques will be used to perform the roughness character-
321 ization of fractured cement pastes. It also seems pertinent that extensive studies are required using CSI and
322 “window resizing” to better understand fractal features of fractured cement-based materials such as cement
323 pastes. The relationships between roughness numbers (and/or fractal dimension) and the mechanical properties
324 (compressive strength, Young modulus, toughness and unit failure work) could then be studied. The effect of
325 the curing time on both polished and unpolished cement pastes could also be considered.

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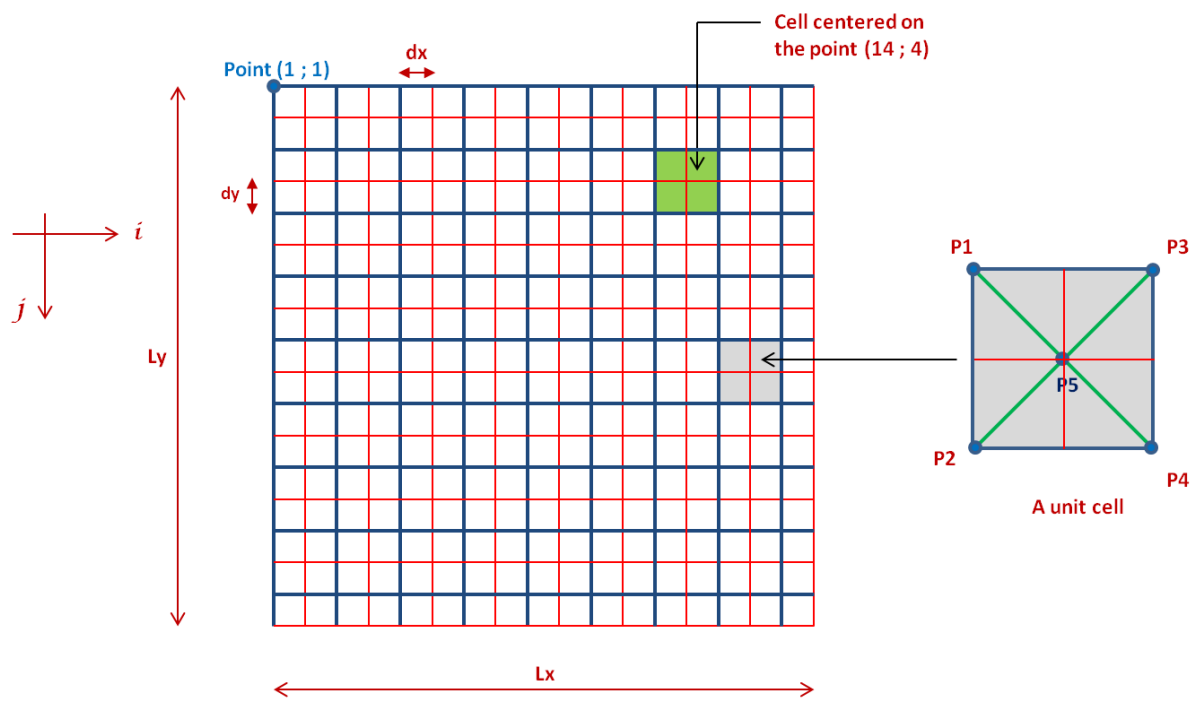
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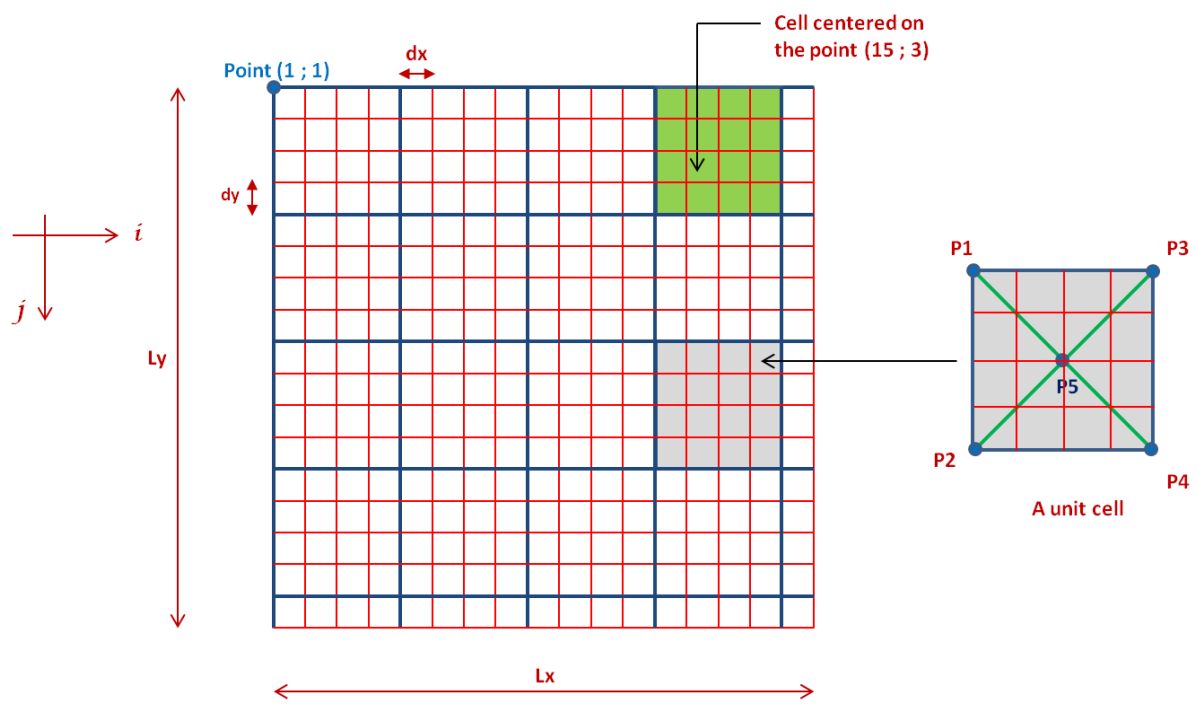
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(a)



(b)

Figure 1: Decomposition into cells (a) : for $\delta = 1$; (b) : for $\delta = 2$.

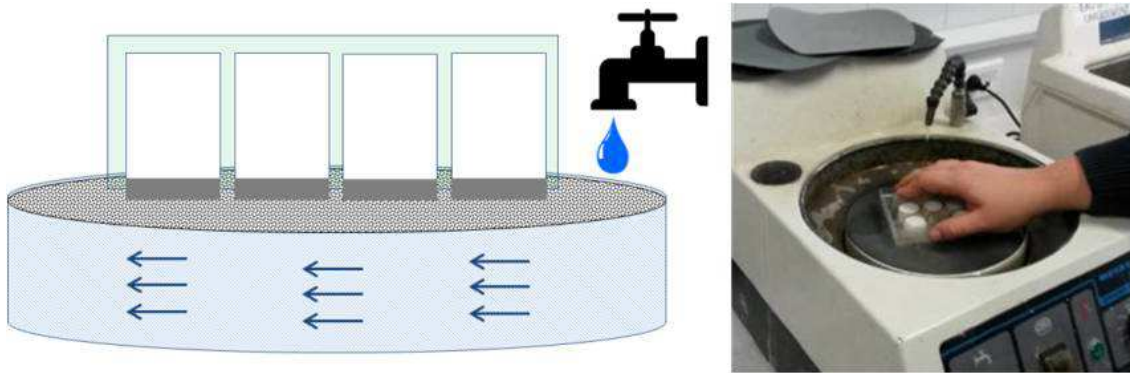
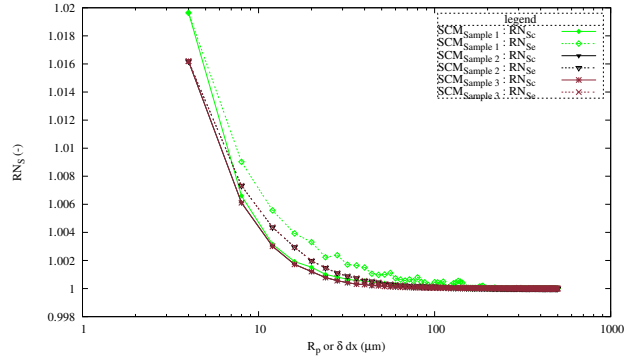
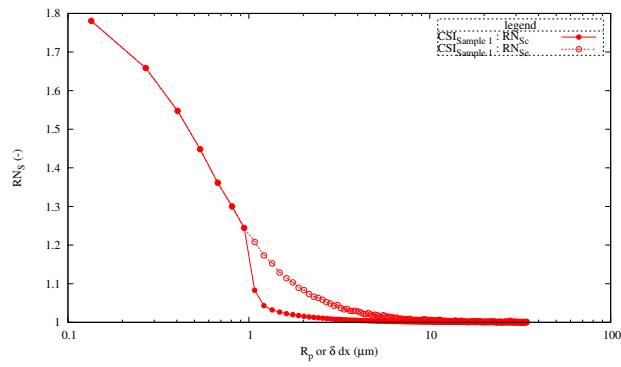


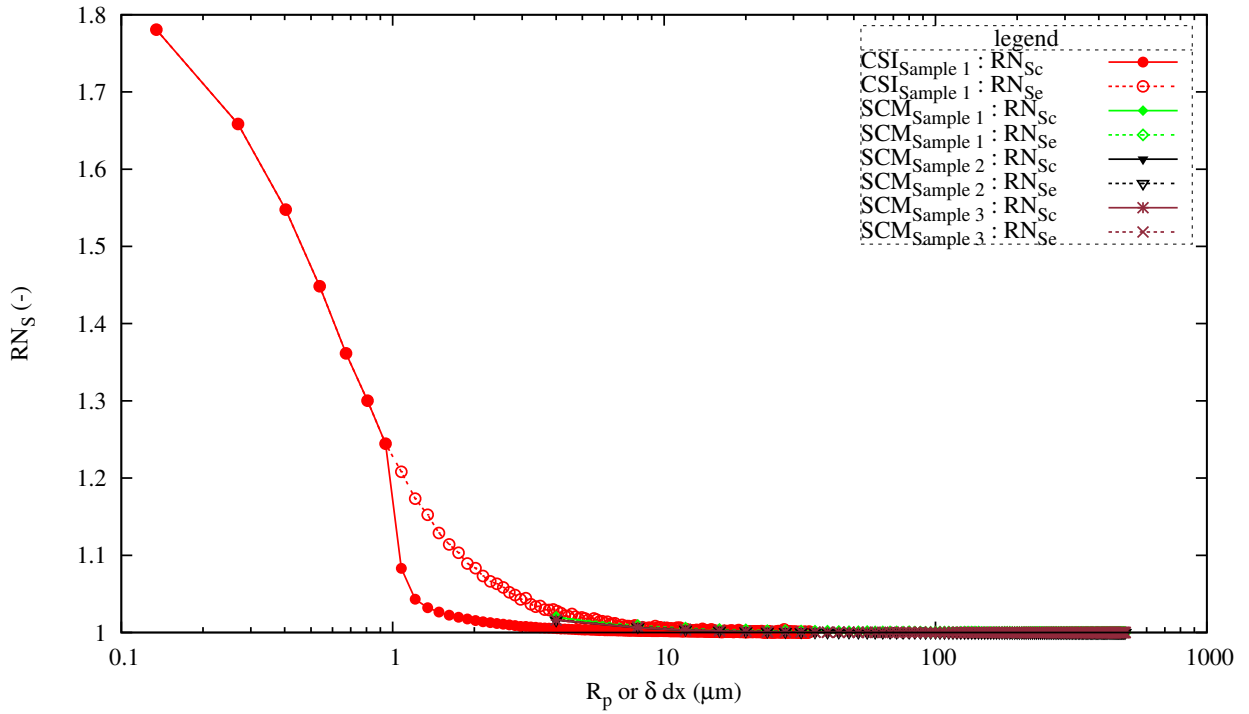
Figure 2: The polishing process.



(a)



(b)



(c)

Figure 3: Roughness number RN_S : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI

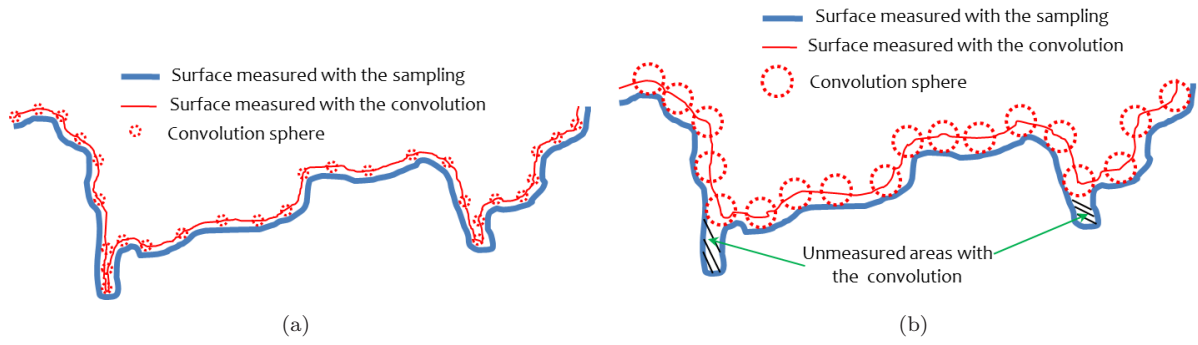
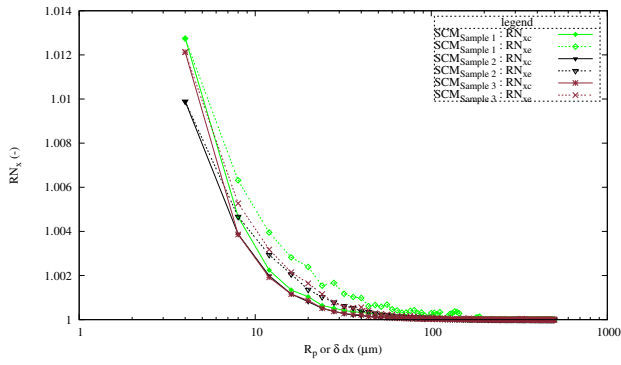
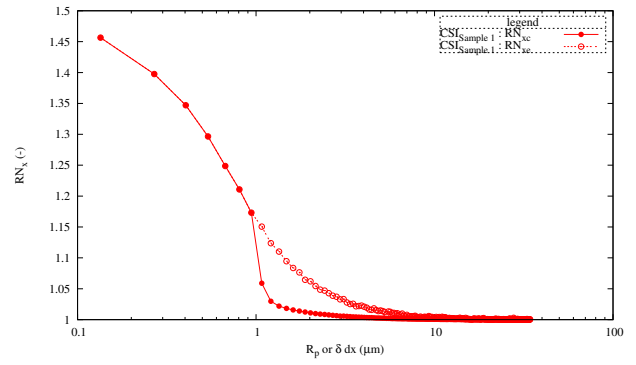


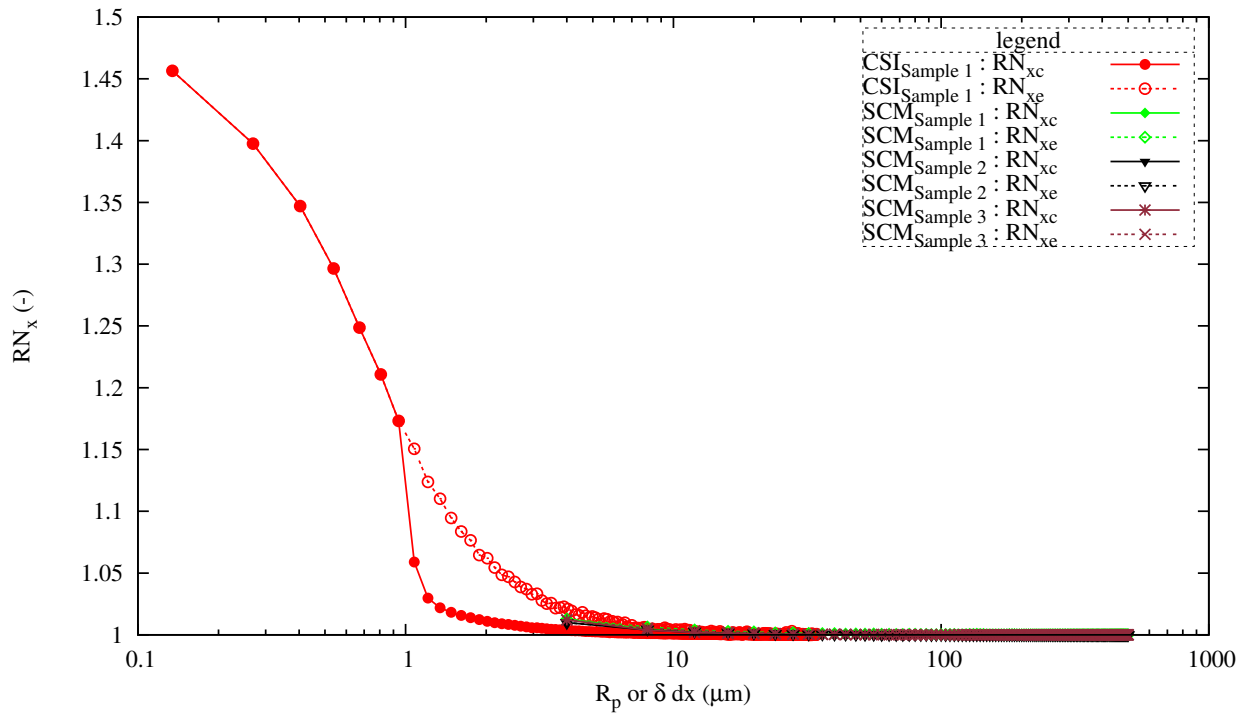
Figure 4: Measured surfaces using CSI (sampling and convolution) : (a) $\delta \leq 8$, (b) $\delta > 8$



(a)

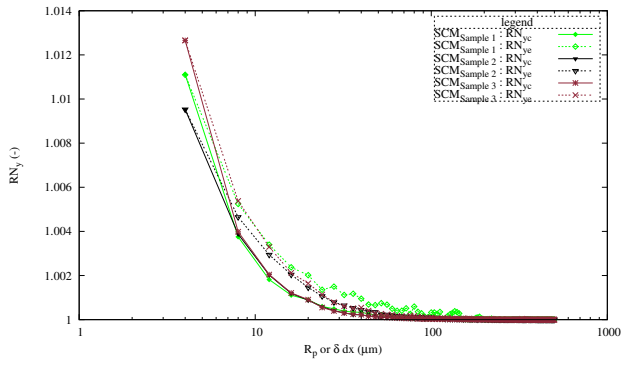


(b)

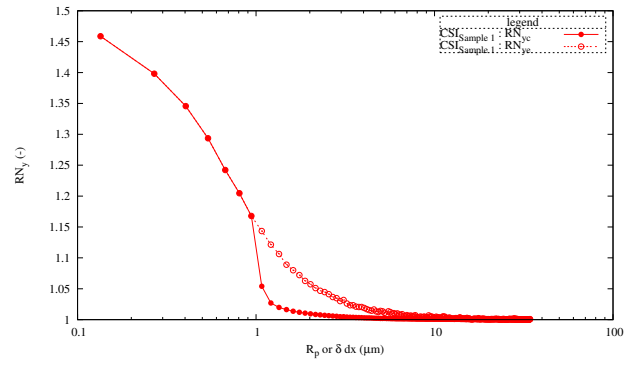


(c)

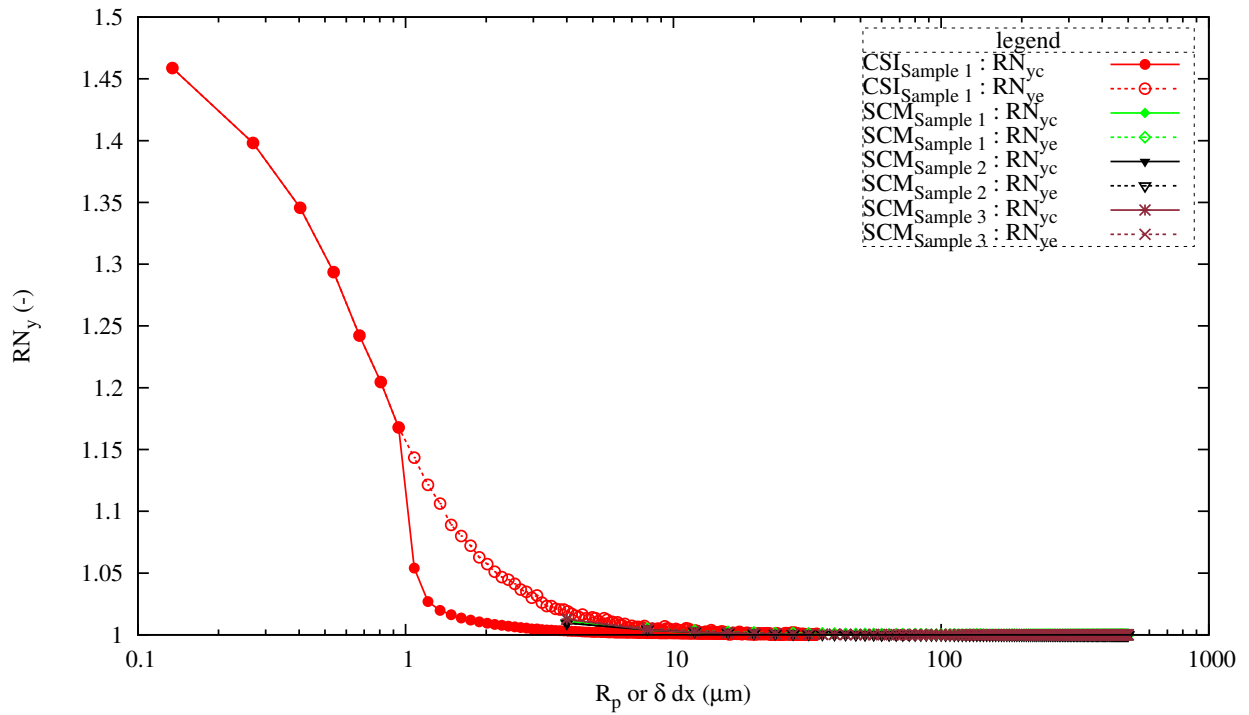
Figure 5: Roughness numbers along x axis : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI



(a)



(b)



(c)

Figure 6: Roughness numbers along y axis : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI

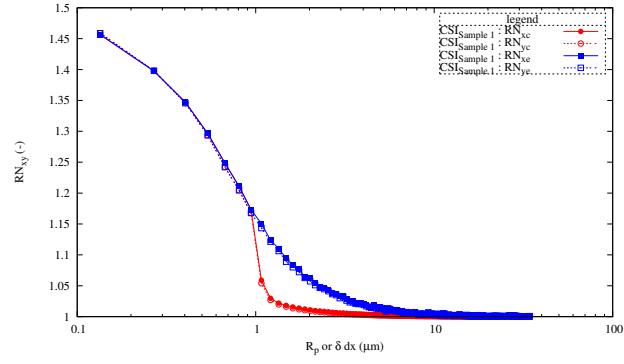
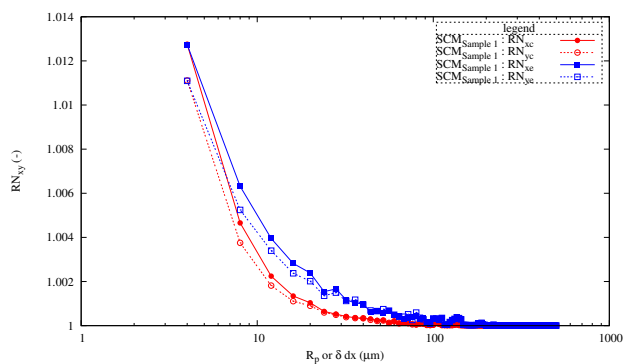
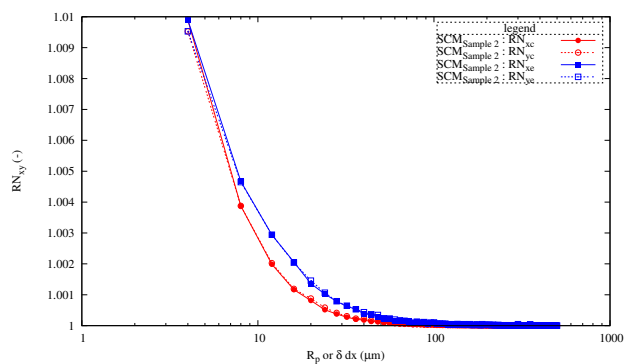


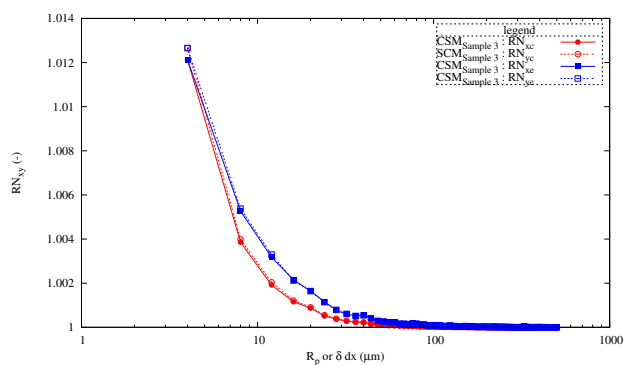
Figure 7: Roughness numbers along both axes in the case of CSI measurements.



(a)

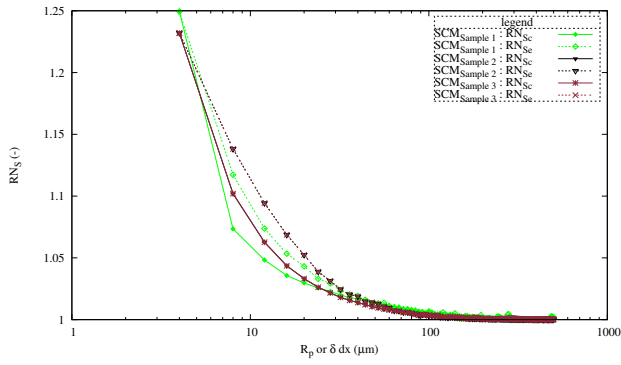


(b)

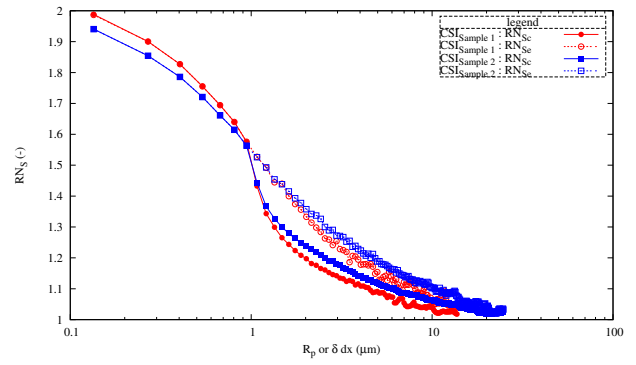


(c)

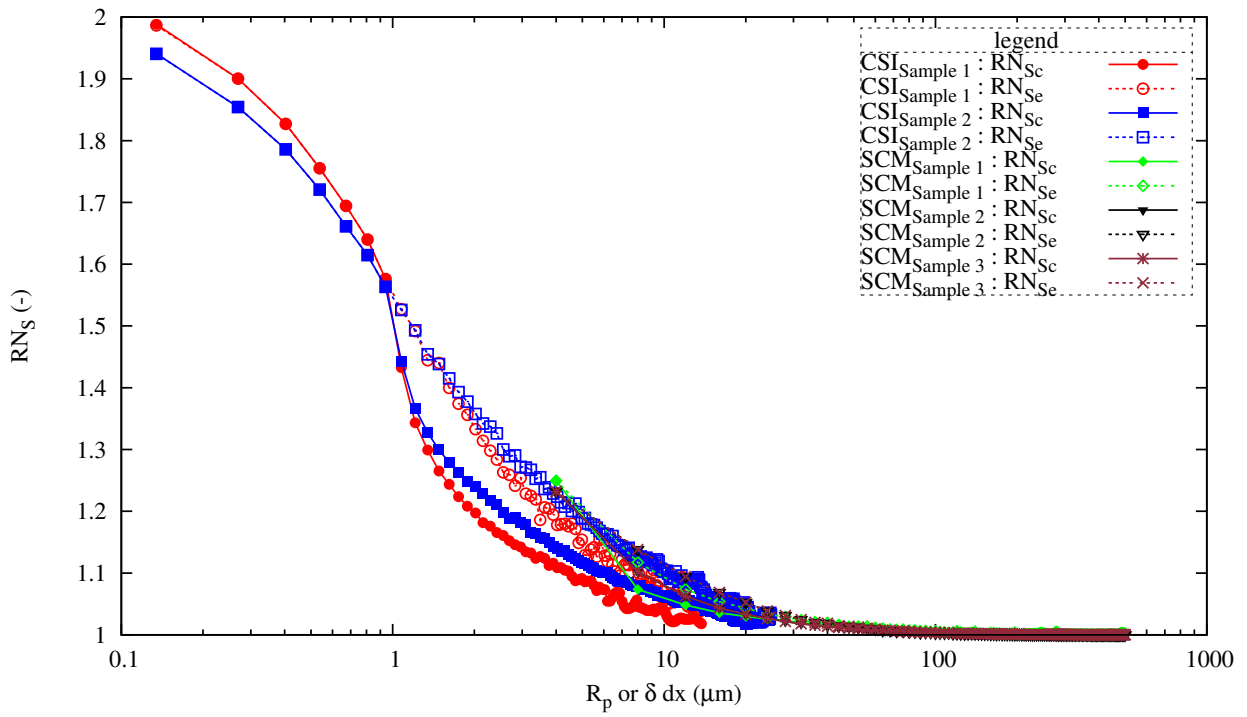
Figure 8: Roughness numbers along both axes in the case of SCM measurements : (a) results from Sample 1, (b) results from Sample 2, (c) results from Sample 3



(a)



(b)



(c)

Figure 9: Roughness RN_S numbers in the case of unpolished samples : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI

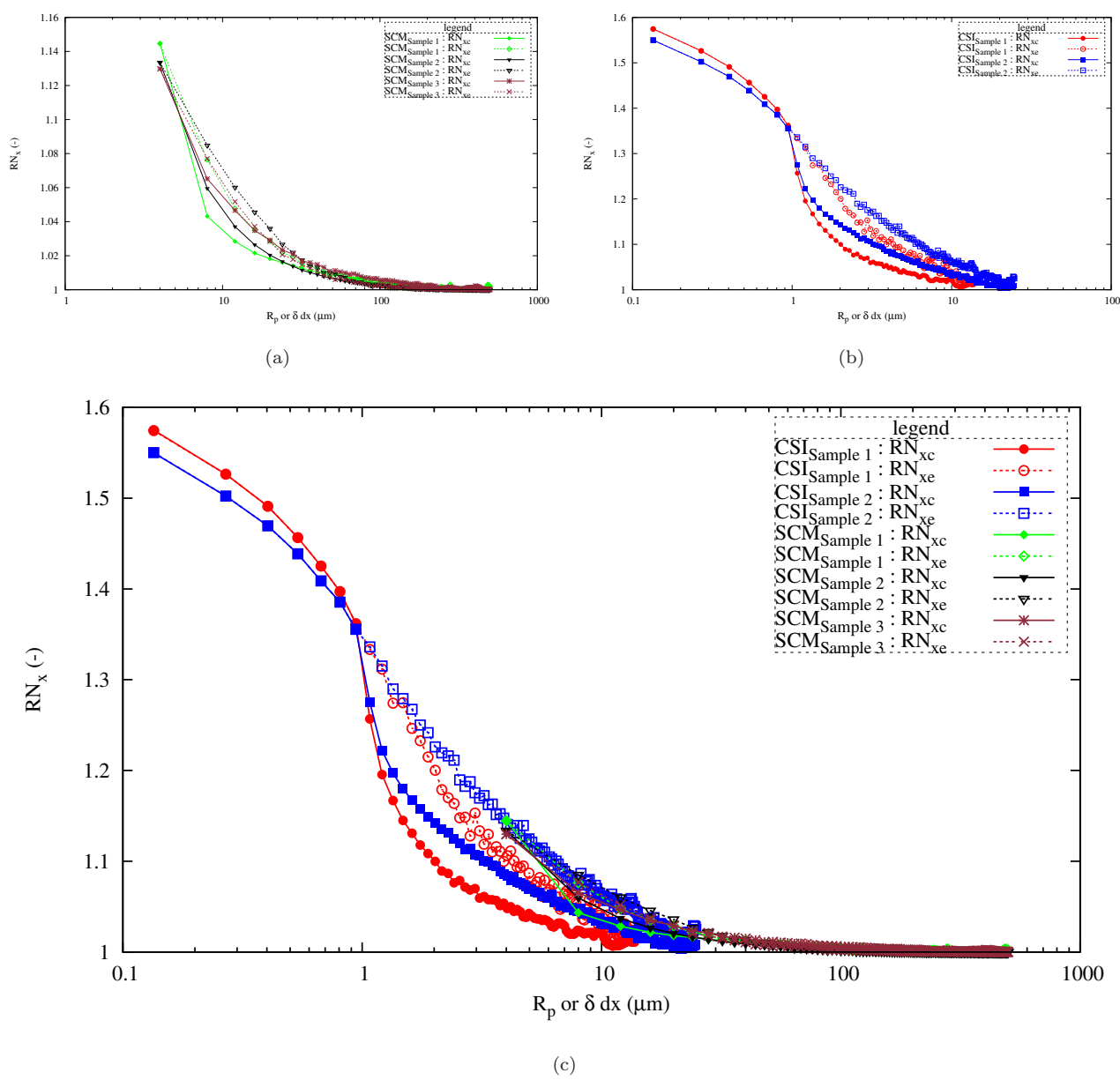
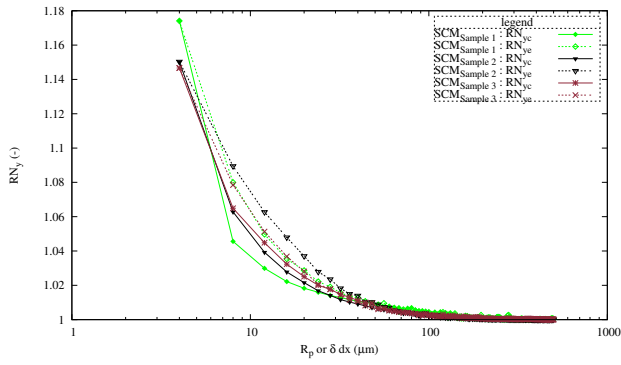
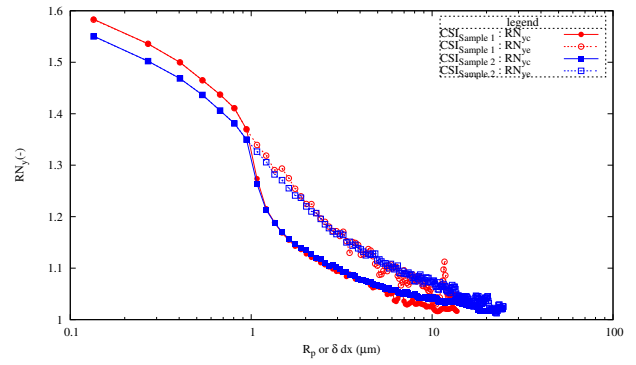


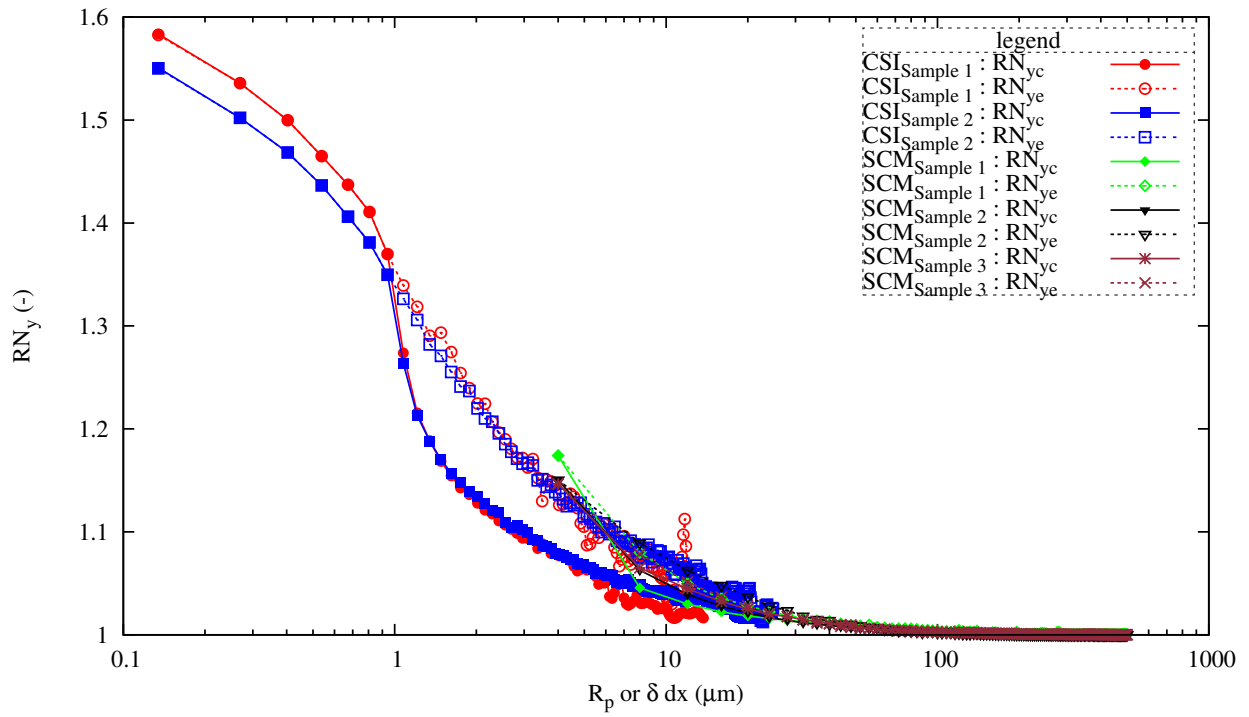
Figure 10: Roughness RN_x numbers along x axis in the case of unpolished samples : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI



(a)

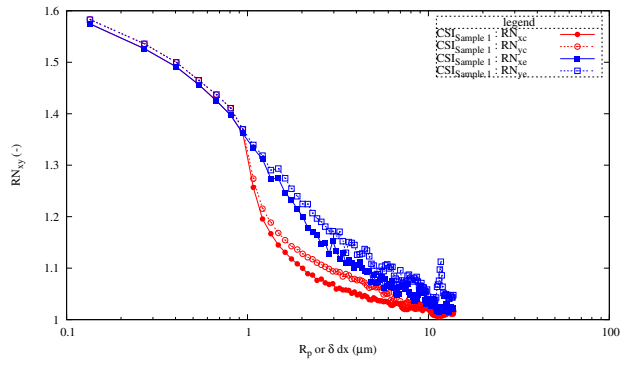


(b)

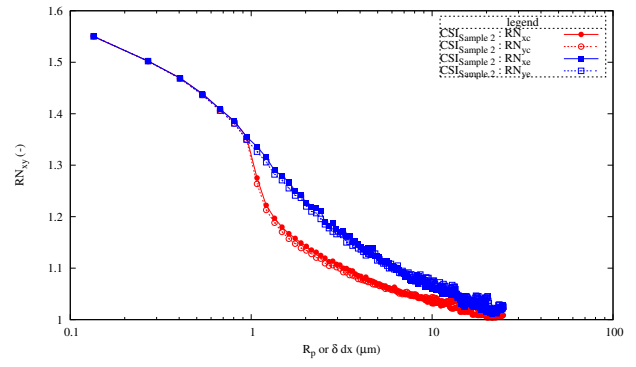


(c)

Figure 11: Roughness RN_y numbers along y axis in the case of unpolished samples : (a) results from SCM, (b) results from CSI, (c) results SCM vs CSI

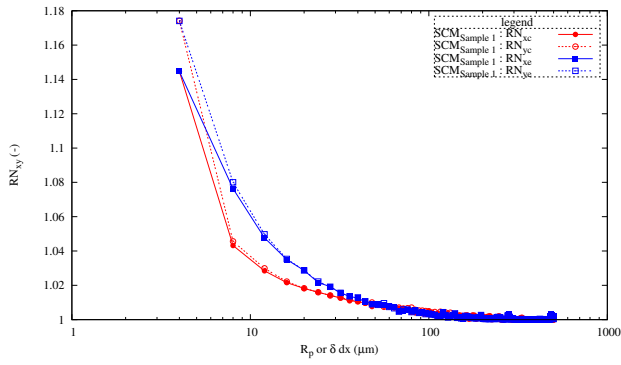


(a)

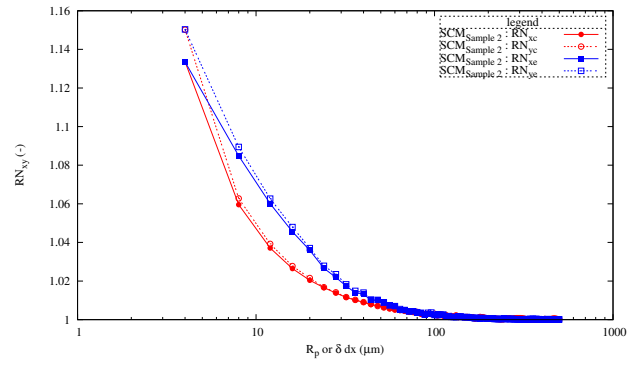


(b)

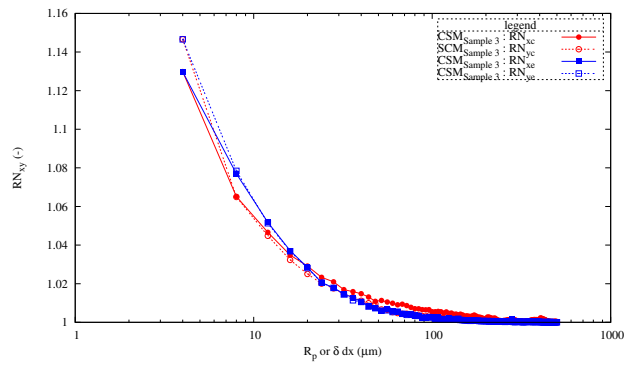
Figure 12: Roughness numbers along both axes in the case of CSI measurements : (a) results from Sample 1, (b) results from Sample 2



(a)



(b)



(c)

Figure 13: Roughness numbers along both axes in the case of SCM measurements : (a) results from Sample 1, (b) results from Sample 2, (c) results from Sample 3