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1 **Quantity and quality of dispersed fine particles after low-energy water-dispersible**
2 **soil test: impact of the initial soil matrix potential**

3

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10 **Abstract**

11 Transfer of $<2 \mu\text{m}$ soil particles is a key process in the dispersion of pollutants and the textural
12 differentiation of soil. Dispersion tests involving low-energy water-dispersible clay (LEWDC)
13 are commonly used to indicate the stock of particles $<2 \mu\text{m}$ that can be mobilized. However,
14 notable differences in LEWDC protocols exist. We discuss the impact of the initial soil matrix
15 potential on the dispersion sensitivity of $<2 \mu\text{m}$ particles and the nature (mineralogy and particle
16 size) of the dispersed particles. The dispersion sensitivity was more differentiated among soils
17 with a high initial water content, which allows better soil discrimination. The nature of dispersed
18 $<2 \mu\text{m}$ particles was closer to that of the bulk $<2 \mu\text{m}$ soil when moist initial conditions are
19 considered, whereas enrichment in coarse clay minerals (with a particle size mode around $2 \mu\text{m}$)
20 was observed with dry initial conditions. Performing the LEWDC test under both moist and dry
21 initial conditions can be used to interpret soil functioning.

22

23 Abbreviations: LEWDC: low-energy water-dispersible clay

24

25 **Highlights**

- 26 - Moist initial condition allows better discrimination of particle dispersion among soils
- 27 - Nature of dispersed <2 μm particles varies with the initial soil matrix condition
- 28 - LEWDC test at two different initial conditions allows soil functioning analysis

29

30 **Acknowledgments**

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37 authors are grateful to Arvalis for providing access to their long-term experimental site.

38

39

40 **Supplemental file:**

41 S1: Concentration of LEWDC dispersed <2 μm particles (LEWDC in $\text{g}\cdot\text{kg}^{-1}$) for two the initial matrix
42 potentials.

43

44

45

46 **1 Introduction**

47 Transfer of $<2 \mu\text{m}$ soil particles is a key process in the dispersion of pollutants, as the fine
48 particles act as vectors for many pollutants (McCarthy and Zachara, 1989), and in the textural
49 differentiation of soil (Pedro et al., 1978). However, depending on the nature of the particles
50 various types of contaminants could be adsorbed and transported, as previously demonstrated for
51 radionuclides, metals, pesticides, and bacteria (e.g., McCarthy et al., 1998; Karathanasis, 1999;
52 Gouy et al., 1999; Ryan et al., 1999, respectively). In addition, textural differentiation of soil is
53 considered to result from a preferential transport of either smectites (e.g., Worrall et al., 1999) or
54 other mineralogical clays (Cornu et al., 2014). Thus, determining the nature of dispersed particles
55 is the first step in assessing the environmental impacts in such systems.

56 In parallel, laboratory dispersion tests have been developed to estimate soil aggregate stability or
57 soil erodibility (Brubaker et al., 1992; Emerson, 1967; Le Bissonnais, 1996; Pojasok and Kay,
58 1990; Yoder, 1936). A large variety of dispersion protocols exist and have been extensively
59 reviewed by Amézketa (1999). These tests were also used as indicators of the stock of $<2 \mu\text{m}$
60 particles that can be mobilized from a given soil, as suggested by Kjaergaard et al. (2004) or,
61 Dexter et al. (2011) and could thus also be used to determine the nature of the mobilized
62 particles, i.e., their mineralogy and size. However, these dispersion tests have never been used for
63 the latter determination to our knowledge. Kjaergaard et al. (2004) showed that the use of low-
64 energy during the dispersion phase better represents the stock of particles less than $2 \mu\text{m}$ that can
65 be dispersed compared to the protocol based on a classical high mechanical energy. These
66 authors proposed a “low-energy water-dispersible clay” (LEWDC) protocol that may mimic in
67 situ particle mobilization from the upper soil horizon. Dexter et al. (2011) confirmed the
68 importance of the initial matrix potential on the result of this test and underscored the benefit of

69 using two initial matrix potentials: a low potential pF 2.5 [-30 kPa] to mimic soil conditions at the
70 end of a humid period and a high potential pF 6 [-100000 kPa] to mimic the dry topsoil at the
71 surface.

72 In that context, our objective was to identify the nature of the particles released by a low-energy
73 dispersion test considered as a proxy for clay transfer in soils and to determine if the initial
74 matrix potentials of the soil act on the nature (mineralogical composition and size distribution) of
75 the dispersed particles. For that purpose, we propose here a LEWDC adapted to address this
76 objective.

77

78 **2. Materials and Methods**

79 2.1 Studied soils and total soil analysis

80 Seven arable soils were sampled from the A-horizon for 6 of them (Villamblain, Boigneville A,
81 Boigneville B, Brosse, Cercottes, Mont-près-Chambord, and Faux-Perche) and from the E-
82 horizon (Brosse) for the latter. Boigneville A and B were two plots of the same soil that
83 experienced different agricultural practices, resulting in a higher organic carbon (OC) content in
84 Boigneville B. According to the USDA texture classification, most soils had a silty loam texture
85 ranging from silty clay loam and silt loam, except for Cercottes and Mont-près-Chambord, which
86 had sandy loam and sandy clay textures, respectively (Table 1). These soils were chosen to
87 represent a large span of content of $<2 \mu\text{m}$ particles (from 12 to 37%) and organic carbon content
88 (0.3 to 3%) with a focus on loamy soils that are considered as the most sensitive soils to clay
89 transfer (Pedro et al., 1978). Their cation exchange capacity (CEC) ranged from 77 to 309
90 $\text{mmol}^+ \text{kg}^{-1}$, and their pH ranged from 5.4 to 8.3. More than 80% of their CEC was due to Ca^{2+}
91 and less than 2.5% to Na^+ . For each soil, large undisturbed soil blocks were cut off the soil, along

92 a pit, with a knife and stored in box (30cm x 20cm x 15cm) at 4 °C before LEWDC test. Depth of
93 sampling was 0-10 cm, except for the Brosse site where sampling took place between 35 and 45
94 cm depth.

95 Particle size distribution was determined on the whole soil according to the normalized protocol
96 NF X 31-107 (by sieving and the Robinson pipette method); water pH, according to the
97 normalized protocol NF ISO 10390; CEC and exchangeable cations, using the cobaltihexamine
98 chloride method according to the normalized protocols NF ISO 23470 and NFX 31-130,
99 respectively; and OC, according to the normalized protocol NF ISO 10694 with combustion at
100 1000°C.

101

102 2.2 Dispersion test protocol

103 LEWDC protocol consists of 3 steps which vary among authors. We built up our own protocol
104 based on the most used settings.

105 • Pretreatment: The soil samples were equilibrated at a given matrix potential to control
106 their water content. Two pretreatments were chosen: “pF 2.5” [-30 kPa] to obtain soil samples
107 close to field capacity and “pF 6” [-100000 kPa] to obtain soil samples with a water content
108 close to that of the soil surface during summer. The whole undisturbed soil samples were
109 manually split into centimeter-size clods, saturated by capillarity on foam for 24 hours, and
110 placed in a Richards apparatus (Richards, 1956) for 7 days for equilibration at a [-30 kPa]
111 water potential for pF 2.5 [-30 kPa]. For pF 6 [-100000 kPa], the disturbed soil samples were
112 oven dried at 35 °C for 5 days.

113 • Wetting: Two approaches exist in the literature, either immediate immersion after
114 pretreatment (Emerson, 1967; Le Bissonnais, 1996; Seta and Karathanasis, 1996; Dexter et

115 al., 2011; Vendelboe et al., 2012) or slow resaturation (Kjaergaard et al., 2004). This last
116 pretreatment being less common we chose an immediate immersion after pretreatment: the
117 clods were placed in a glass bottle with osmosed water at a soil:water ratio of 1:8 (ratio
118 classically used in the literature).

119 • Dispersion: For low energy dispersion the most classical approach consists in a ten fold
120 manual inversion (Kjaergaard et al., 2004, Vendelboe et al., 2011). In this study the soil
121 suspension in the glass bottle was manually inverted 10 times, over 1 minute. Then, the
122 suspension was allowed to settle for 6 hours and 14 minutes for collection of $<2 \mu\text{m}$ particle
123 according to Stokes law, the upper 8cm were then collected.

124

125 2.3 Analysis of dispersed $<2 \mu\text{m}$ particles

126 The particle concentrations of the LEWDC suspensions were measured by turbidimetry (Hach
127 model 2100AN, Hach Compagny, Loveland, CO) after calibration. The uncertainty of the
128 measurements was estimated at 5%.

129 Particle size was analyzed by a laser particle sizer (Malvern S) after further dispersion by NaPO_3
130 addition. The mineralogical composition of the $<2 \mu\text{m}$ particles was determined by X-ray
131 diffraction (XRD; Bruker-AXS, D8 Advance) on oriented slides by deposition of 1 mL of a
132 7.5 g L^{-1} suspension that was air dry; samples were analyzed after the application of two
133 treatments, following the methodology of Robert and Tessier (1974): air drying and ethylene
134 glycol (EG) solvation.

135 The particle size and mineralogy were measured for (1) the $<2 \mu\text{m}$ fraction of the bulk soils
136 obtained by the Robison's pipette method and (2) the $<2 \mu\text{m}$ fraction obtained after the LEWDC
137 tests at the two initial matrix potentials. The nature of the dispersed particles at the Mont-près-
138 Chambord and Cercottes sites was not analyzed due to limited sample quantities.

139
140 2.4 Data treatment: statistical analysis
141 For each LEWDC test replicates were performed. Results were presented with mean and standard
142 deviation except for XRD (all replicates presented).
143 For each site, an ANOVA at the 5% confidence level (Fisher pair-wise mean comparison
144 method) was used to assess the effects of soil properties and initial matrix potential on the
145 amount of LEWDC dispersed particles.

146
147
148 **3 Results**

149
150 3.1 Quantity of dispersed particles at different initial soil matrix potentials
151 The quantity of LEWDC dispersed particles was significantly higher at pF 2.5 [-30 kPa] than at
152 pF 6 [-1000000 kPa] in all the tested soils except for Mont-près-Chambord (Fisher's test at the
153 95% confidence level considering the two initial matrix potential together—Figure 1). The
154 greatest variation between the pF 6 [-1000000 kPa] and pF 2.5 [-30 kPa] pretreatments was
155 observed for Brosse. The difference in particle dispersion among the sites was greater at pF 2.5 [-
156 30 kPa], ranging from 37.2 g.kg⁻¹ to 5.7 g.kg⁻¹, than at pF 6 [-1000000 kPa], ranging from 5.8
157 g.kg⁻¹ to 1.4 g.kg⁻¹.
158 While comparing the quantity of particles LEWDC-dispersed by the different studied soils, i.e.
159 their sensitivity to dispersion, the soils did not follow the same sensitivity sequence for the two
160 initial matrix potential i.e., pF 6 [-1000000 kPa] and pF 2.5 [-30 kPa] (Fisher's test at the 95%
161 confidence level). Nevertheless, for both pretreatments, the site most sensitive to dispersion was
162 Brosse (Supplementary file, S1). Thus, the quantity of dispersed particles at a given matrix
163 potential is not proportional to that at the other matrix potentials.

164
165 3.2 Nature of the LEWDC dispersed $<2\ \mu\text{m}$ particles
166 The nature of the LEWDC particles, defined by their mineralogical composition and particle size
167 distribution, was compared at the two initial matrix potentials and with the nature of the total
168 $<2\ \mu\text{m}$ fraction of the soils for the five following sites: Brosse, Villamblain, Boigneville A,
169 Boigneville B and Faux-Perche.

170

171 3.2.1 Mineralogical composition

172 The LEWDC dispersed $<2\ \mu\text{m}$ particles were richer in quartz at pF6 [-1000000 kPa] than at
173 pF 2.5 [-30 kPa] and at pF 2.5 [-30 kPa] than in the total $<2\ \mu\text{m}$ soil fraction. Concerning clay
174 minerals, three patterns were observed. For Brosse, the LEWDC dispersed $<2\ \mu\text{m}$ particles were
175 poorer in smectite than was the total $<2\ \mu\text{m}$ fraction (Figure 2 a and b). This deficit was greater
176 for the pretreatment at pF 6 [-1000000 kPa] than for that at pF 2.5 [-30 kPa]. For Boigneville A
177 and B and Villamblain, the mineralogical composition of the LEWDC dispersed $<2\ \mu\text{m}$ particles
178 at pF 2.5 [-30 kPa] was not different from that of the total $<2\ \mu\text{m}$ fraction; however, the LEWDC
179 dispersed $<2\ \mu\text{m}$ particles were deficient in smectite relative to the total $<2\ \mu\text{m}$ fraction at pF 6 [-
180 1000000 kPa] (Figure 2c and d—only Boigneville A is presented). For Faux-Perche, the LEWDC
181 dispersed $<2\ \mu\text{m}$ particles had the same mineralogical composition as the total $<2\ \mu\text{m}$ fraction of
182 the soil, irrespective of the initial matrix potentials (Figure 2e and f).

183

184 3.2.2 Particle size distribution

185 The particle size distribution of the total $<2\ \mu\text{m}$ fraction was bimodal for Brosse, Boigneville A
186 and Villamblain, with two modes of similar proportion at approximately $0.2\ \mu\text{m}$ and $2\ \mu\text{m}$
187 (Figure 3a, c, and e). The mode at approximately $0.2\ \mu\text{m}$ is related to the presence of smectite,

188 while the second mode is due to either larger clay minerals (illite, kaolinite, chlorite, quartz, and
189 feldspars) or microaggregates of smectites, as shown by Hubert et al. (2012).

190 For Faux-Perche, the particle size distribution of the $<2 \mu\text{m}$ fraction exhibited a unique mode at
191 approximately $2 \mu\text{m}$ (Figure 3b), which is consistent with the absence of smectite in this soil
192 (Figure 2e and f). For Boigneville B, the particle size distribution of the $<2 \mu\text{m}$ fraction also
193 exhibited a main mode at approximately $2 \mu\text{m}$ but with a slight shouldering at approximately
194 $0.53 \mu\text{m}$ (Figure 3d). The absence of a $0.2 \mu\text{m}$ mode in the particle size distribution may have
195 been due to the presence of microaggregates of smectite stabilized by organic bonds. This is
196 consistent with the higher OC content in Boigneville B than in Boigneville A (Table 1).

197 While comparing the particle size distribution of the LEWDC dispersed $<2 \mu\text{m}$ particles to that of
198 the total $<2 \mu\text{m}$ fraction of the soils, four patterns were observed. For Brosse, the LEWDC
199 dispersed $<2 \mu\text{m}$ particles presented a bimodal particle size distribution comparable to that of the
200 total $<2 \mu\text{m}$ fraction but with slight shifts in the positions of the modes and their proportions
201 (Figure 3a). After the LEWDC test, the proportion of fine particles decreased, while the
202 proportion of coarse particles increased, especially at pF 6 [-1000000 kPa].

203 Regarding Boigneville A and Villamblain, the LEWDC dispersed $<2 \mu\text{m}$ particles pretreated at
204 pF 2.5 [-30 kPa] exhibited a bimodal distribution with a smaller fine particle mode (and larger
205 coarse mode) than that of the total $<2 \mu\text{m}$ fraction, while the LEWDC particles pretreated at pF 6
206 [-1000000 kPa] had a monomodal distribution centered at the same position as the coarse mode
207 of the total $<2 \mu\text{m}$ fraction (Figure 3c and e).

208 Concerning Faux-Perche, the LEWDC dispersed $<2 \mu\text{m}$ particles had a monomodal particle size
209 distribution similar to that of the total $<2 \mu\text{m}$ fraction of the soil but with a slight increase in the
210 mode at pF 6 [-1000000 kPa] (Figure 3b).

211 For Boigneville B, the LEWDC dispersed $<2 \mu\text{m}$ particles exhibited a monomodal distribution
212 comparable to that of the total $<2 \mu\text{m}$ fraction but with a smaller shouldering at pF 2.5 [-30 kPa].
213 At pF 6 [-1000000 kPa], this shouldering disappeared, and the mode was shifted toward coarser
214 particles, with a large proportion of particles $>2\mu\text{m}$ probably due to aggregation processes after
215 sampling (Figure 3d).

216 As smectites are the finest clay mineral particles, the decrease in the $0.2 \mu\text{m}$ mode corresponds to
217 the decrease in smectites observed in the XRD data, while the increase in the $2 \mu\text{m}$ mode
218 corresponds to an increase in other clay minerals, e.g., feldspars and quartz.

219

220 **4. Discussion/Conclusion**

221 Our results demonstrated that the amount of LEWDC dispersed $<2 \mu\text{m}$ particles vary strongly
222 with the initial matrix potential of the sample, and this variation differs in amplitude from one
223 soil to another. As emphasized by Amézketa in 1999, we also support the need for protocol
224 standardization. The air-drying pretreatment is probably easier to standardize; nevertheless, the
225 sensitivity of soils to particle dispersion by the LEWDC test was more pronounced at pF 2.5 [-30
226 kPa], as observed by Dexter et al. (2011). Therefore, to determine the sensitivity of different soils
227 to particle dispersion, we recommend a controlled, moist initial condition equal to pF 2.5 [-30
228 kPa].

229 We showed that the nature (mineralogical composition and particle size distribution) of dispersed
230 $<2 \mu\text{m}$ particles is not similar to that of the total $<2 \mu\text{m}$ fraction and is a function of the initial
231 matrix potential. The nature of the dispersed $<2 \mu\text{m}$ particles was generally more similar to that
232 of the total $<2 \mu\text{m}$ fraction at pF 2.5 [-30 kPa] than at pF 6 [-1000000 kPa] and always
233 accompanied by a slight enrichment in coarser minerals. Depending on the soil type, this particle
234 segregation is more or less pronounced. Therefore, we propose applying the LEWDC test at two

235 contrasting initial matrix potentials with a systematic determination of the nature of the dispersed
236 $<2 \mu\text{m}$ particles in order to interpret the results in terms of soil processes such as soil textural
237 differentiation or soil contamination via particle transfer.
238

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300

301 Table 1: Pedological characteristics of the total soils.

302

303

Site	Particle size (g.kg ⁻¹)			OC (g.kg ⁻¹)	pH _{H2O}	CEC	Ca ²⁺	Mg ²⁺	K ⁺	Na ⁺
	0-2 μm	2-50	50-2000							
Brosse	307	587	106	3.5	6.2	178	155	11	0	4
Villamblain	328	648	24	14.6	7.8	210	186	8.6	5.9	0.6
Boigneville A	295	633	72	12.1	6.9	147	134	6.9	4.3	0.4
Boigneville B	247	657	96	30	5.4	142	116	7.1	8.8	0.5
Faux Perche	125	825	50	11.1	7.9	77	95	4.4	4.3	0.2
Cercottes	197	143	660	12.7	8.2	140	140	4.6	11.9	0.19
Mont-près-Chambord	366	158	476	14.3	8.3	309	278	15.6	10.2	0.4

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OC: organic carbon, CEC: cation exchange capacity

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List of figures

Figure 1: Concentrations of dispersed 0-2 μm particles for the different studied soils for the two pretreatments. Circles correspond to the pF 2.5 pretreatment, and squares to the pF 6 pretreatment. Letters correspond to a Fisher's mean comparison test performed for all the samples with the two pretreatments at the 95 % confidence level.

Figure 2: XRD patterns of the 0-2 μm fractions of some of the studied soils: a and b—Brosse, c and d—Boigneville A, and e and f—Faux Perche. a, c and e—air dry treatment; b, d and f—EG solvation. For each site, we present the total 0-2 μm fraction of the soil (black) and the 0-2 μm fraction extracted in the LEWDC tests at pF 6 (red) and pF 2.5 (blue). Multiple curves of the same color represent replicates.

Figure 3: Particle size distribution of the 0-2 μm fractions of the studied soils: a) Brosse, b) Faux Perche, c) Boigneville A, d) Boigneville B and e) Villamblain. For each site, the total 0-2 μm fraction (black) and the 0-2 μm particles dispersed in the LEWDC test at pF 6 (red) and pF 2.5 (blue) are reported.

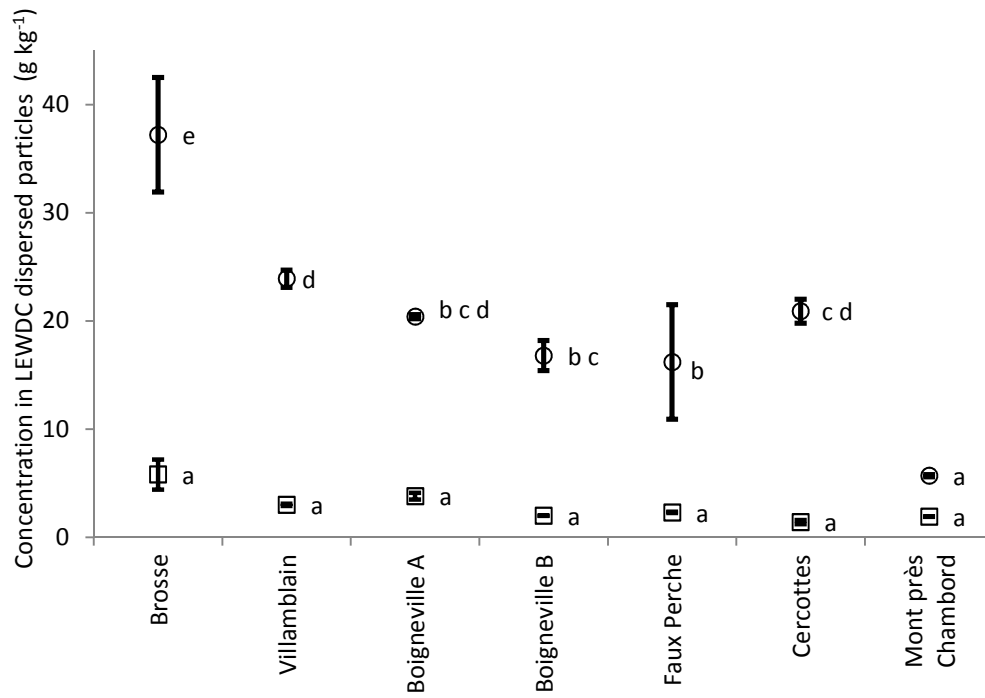


Figure 1: Concentrations of dispersed 0-2 μm particles for the different studied soils for the two pretreatments. Circles correspond to the pF 2.5 pretreatment, and squares to the pF 6 pretreatment. Letters correspond to a Fisher's mean comparison test performed for all the samples with the two pretreatments at the 95 % confidence level.

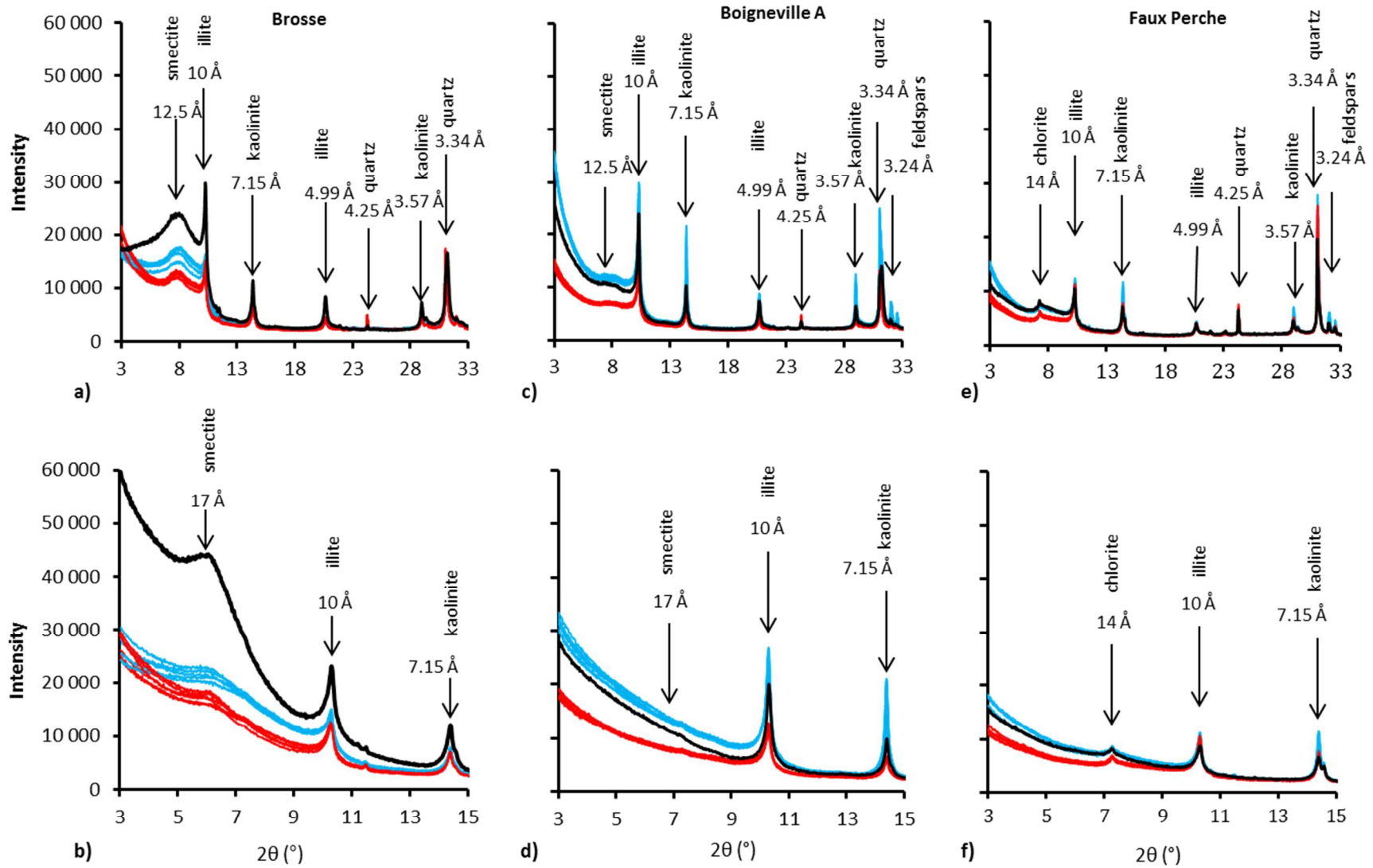


Figure 2: XRD patterns of the 0-2 μm fractions of some of the studied soils: a and b—Brosse, c and d—Boigneville A, and e and f—Faux Perche. a, c and e—air dry treatment; b, d and f—EG solvation. For each site, we present the total 0-2 μm fraction of the soil (black) and the 0-2 μm fraction extracted in the LEWDC tests at pF 6 (red) and pF 2.5 (blue). Multiple curves of the same color represent replicates.

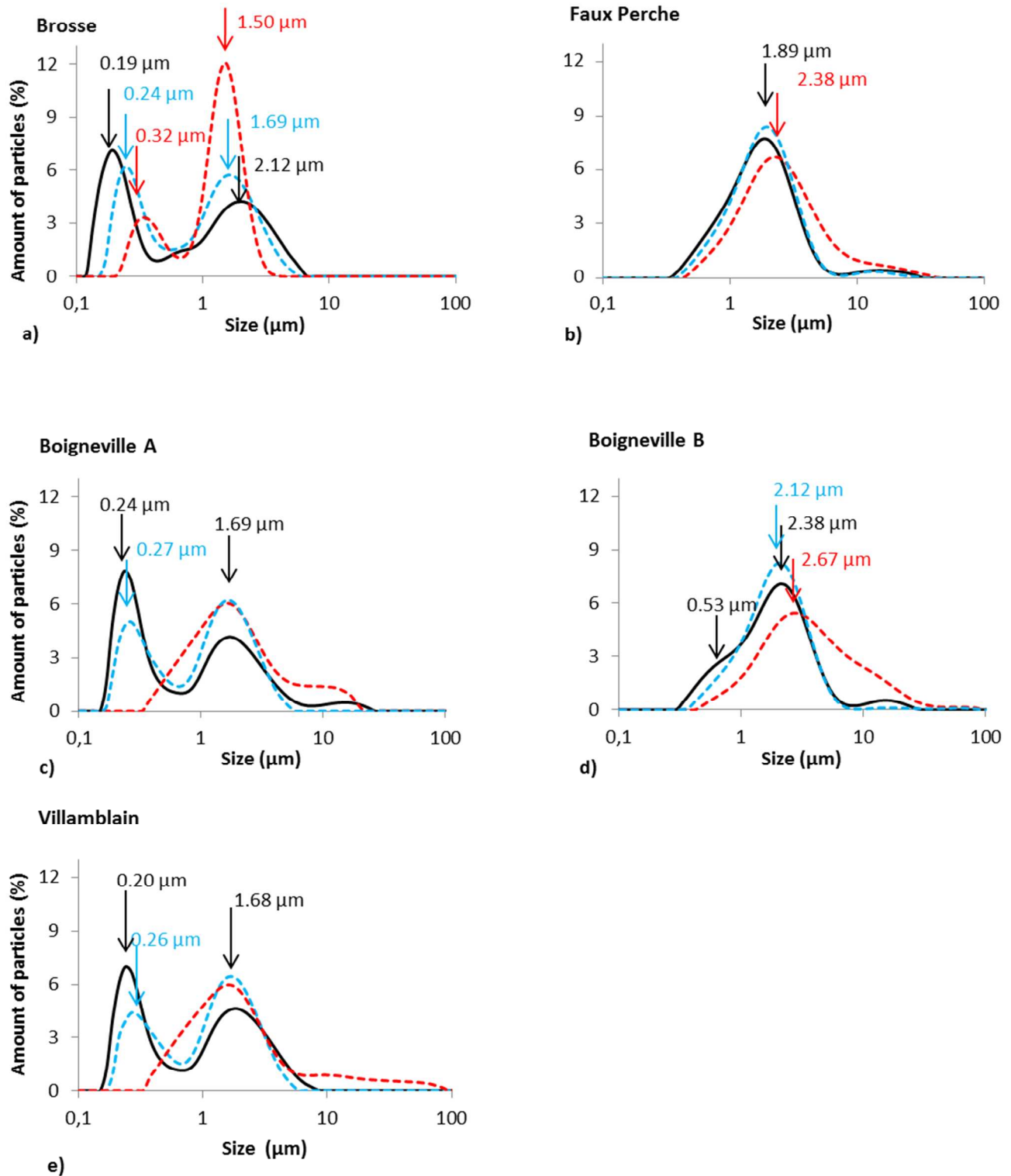


Figure 3: Particle size distribution of the 0-2 μm fractions of the studied soils: a) Brosse, b) Faux Perche, c) Boigneville A, d) Boigneville B and e) Villamblain. For each site, the total 0-2 μm fraction (black) and the 0-2 μm particles dispersed in the LEWDC test at pF 6 (red) and pF 2.5 (blue) are reported.