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## Self-Limited Accumulation of Colloids in Porous Media

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We present local direct imaging of the progressive adsorption of colloidal particles inside a 3D model porous medium. By varying the interparticle electrostatic interactions, we observe a large range of particle deposition regimes, from a single layer of particles at the surface of the medium to multiple layers and eventually clogging of the system. We derive the complete deposition dynamics and show that colloid accumulation is a self-limited mechanism towards a deposited fraction associated with a balance between the particle interactions and the imposed flow rate. These trends are explained and predicted using a simple probability model considering the particle adsorption energy and the variation of the drag energy with evolving porosity. This constitutes a direct validation of speculated particle transport mechanisms, and a further understanding of accumulation mechanisms.

DOI:

Colloidal particles from industrial or natural sources propagate and alter the environment they flow through. Common problems include the accumulation of particles impacting industrial [1,2] or biological [3–6] processes (filtration, storage, cleaning, sorting, etc.) or the leaking of contaminants in ground water [7–9]. Predicting particle transport and stoppage in these porous media is key to solve these problems. All stoppage events have either a geometrical (i.e., straining [10], clogging [11], bridging [12]) or a physicochemical origin (i.e., adsorption). Although the elementary processes are well identified, their development in a porous medium is often described by empirical models. Moreover, because of the complexity of real 3D porous structures, the dynamics of particle deposition are usually extrapolated from indirect observations like breakthrough curves [13–15], direct observations in simplified systems (2D and/or at pore scale) [16–19], and qualitative static imaging or indirect imaging in realistic systems [20,21].

For attractive interactions between suspended particles and porous matrices, several regimes are speculated but unequally understood. First, a particle following a streamline close enough to a surface can be intercepted and adsorbed. Subsequent adsorption is then limited by the presence of previously adsorbed particles, and by interparticle repulsion [22]. This leads to the formation of a uniform single layer of adsorbed particles well described by a simple deposition model relying on a blocking function [23,24] and the DLVO theory [25,26]. Then, for weak interparticle repulsion, particles can adsorb to each other and form complex deposits not limited to the surfaces. As deposits can then grow to larger sizes they may induce significant changes to the flow and boundary conditions, which makes the system much more difficult

to study. These situations are generally ignored or described assuming no coupling between the deposition mechanisms and the state of the flow [27].

Here, we present a full description—by direct internal observation in a model system—of the successive states of deposition from the formation of a monolayer of particles to multilayer accumulation and potential clogging. We explore how the deposition dynamics are affected by the coupled evolution of the pore space and hydrodynamics. Finally, we show that the accumulation of particles can be a self-limited phenomenon driven by a competition between the drag force applied on the adsorbing particles and the colloidal interaction energy.

As a model porous medium we use monodisperse borosilicate beads (negatively charged, 63  $\mu\text{m}$  average diameter) randomly packed (porosity  $\phi_0 = 0.38$ ) in a square glass capillary (width  $w = 1.0$  mm), and we invade this medium with positively charged, fluorescent, monodisperse (diameter  $d_p = 1.0$   $\mu\text{m}$ ) latex colloids. The suspensions are made at constant dilute concentration  $C_0 = 20$   $\text{mg mL}^{-1}$  in a mix of deionized water and DMSO to match the refractive index of the beads [28]. This allows us to visualize the particles inside the porous medium by confocal microscopy. The opposite charges of the beads and the particles fosters particle adsorption at the surface of the beads. Particle-particle bonding may be promoted by screening the interparticle repulsion (i.e., decreasing the Debye length) through an increase of the ionic strength  $I$  by addition of salt (sodium iodide) [29].

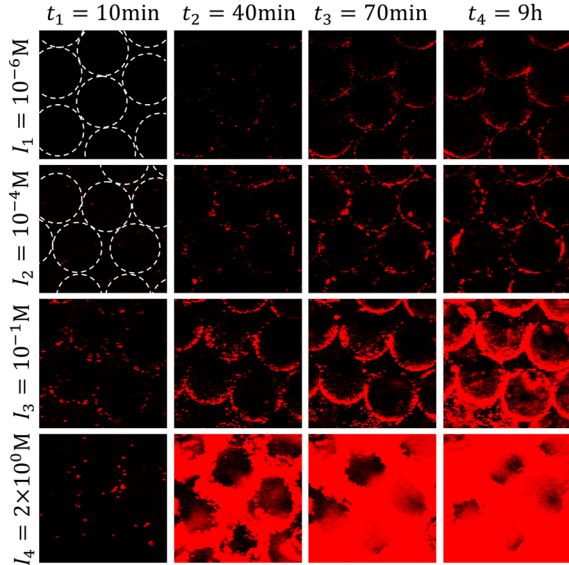
A test consists of continuously injecting a suspension in the porous medium (initially saturated with liquid) and tracking the particle deposition inside the medium. Except when mentioned, we impose a constant flow rate

84  $Q_0 = 5 \mu\text{L min}^{-1}$ , which implies that the local mean  
 85 velocity, i.e.,  $v_0 = Q_0/w^2\phi_0$ , increases when the porosity  
 86 decreases. The flow is laminar: the Reynolds number  
 87 at the maximum flow rate used in this study is  
 88  $\text{Re} = v_0\rho l/\eta = 1.4 \times 10^{-3} \ll 1$ , with  $l = 15 \mu\text{m}$  the typical  
 89 pore scale,  $\eta = 2.5 \text{ mPa s}$  and  $\rho = 1.1 \text{ kg m}^{-3}$  the viscos-  
 90 ity and density of the mix. At some distance from the  
 91 entrance, the concentration of suspended particles may  
 92 significantly vary as a function of the history of adsorption  
 93 all along the porous medium. Here we focus on the  
 94 processes occurring around the entrance of the medium  
 95 (i.e., over a distance of a few bead diameters), where the  
 96 impact of adsorption history is expected to be negligible, so  
 97 that the concentration of suspended particles is considered  
 98 constant and equal to the injected concentration [29].

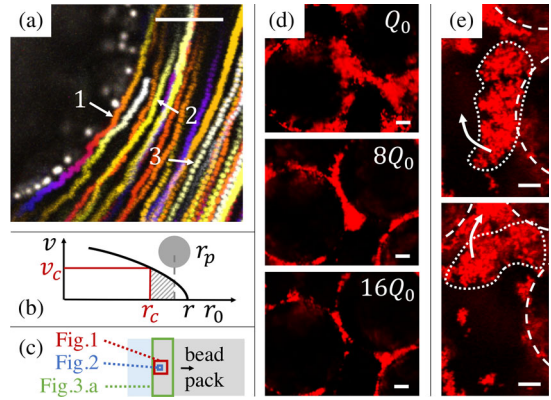
99 Let us first focus on negligible interparticle bonding  
 100 due to strong repulsive electrostatic interactions (i.e.,  
 101  $I = 10^{-6} \text{ M}$ ). Over time, the beads are progressively  
 102 covered by particles adsorbed to their surface (see  
 103 Fig. 1,  $I_1$ - $t_2$ ). A steady state is reached after 1 h of injection  
 104 and characterized by a single layer of particles, outlining  
 105 the surface of the beads (see Fig. 1,  $I_1$ - $t_3$  and  $t_4$ ). The  
 106 coverage is uneven, with a fairly uniform, significant  
 107 coverage on the upstream side of the beads but a negligible  
 108 one on the downstream side. For a single bead in a uniform,  
 109 laminar, fluid flow (far from the bead), the streamlines are  
 110 symmetrical [30] with regards to a cross section.  
 111 Extrapolating this situation to the flow around packed  
 112 beads on average we deduce that a particle following a  
 113 streamline getting closer from the surface than its own  
 114 radius will theoretically first intercept the surface of

the upstream bead face. The collision leads to a surface  
 adsorption event, or a bouncing of the particle to a nearby  
 streamline. This means than the observed preferential  
 adsorption on the elements of surface facing the flow is  
 related to the finite size of the particles.

We also observe that the trajectories of particles are  
 rather smooth at relatively large distance from the walls but  
 become more fluctuating at the approach of some wall [see  
 Fig. 2(a)]. It is worth emphasizing that such effects do not  
 result from inertia effects (small Re) or from diffusion  
 due to thermal agitation, as the Peclet number (i.e.,  
 $\text{Pe} = d_p v_0/D$ , with  $D = 10^{-13} \text{ m}^2 \text{ s}^{-1}$  the diffusion coef-  
 ficient of the particles) is over 100. Smooth trajectories  
 would be obtained for a single particle moving through a  
 simple bead packing [29]. This effect therefore likely  
 results from boundary conditions continuously evolving  
 due to other particles in suspension and depositions at the  
 wall, which in particular rapidly changes the apparent bead  
 roughness. A particle will be even more sensitive to these  
 effects as it is closer to the wall, which explains these  
 apparent fluctuations in the trajectories growing as the  
 particles approach the wall. This likely enhances the  
 adsorption of particles at the bead surfaces, as it tempo-  
 rarily places them closer to the walls. These fluctuations  
 significantly affect the frequency of attempt of adsorption,  
 which depends in a complex way on the particle concen-  
 tration, the flow rate, and the current structure of the  
 deposit. Here, as a critical aspect of our approach, we  
 will consider this frequency as a constant factor for a



F1:1 FIG. 1. Confocal microscopy images of particles (red) deposited  
 F1:2 in the pore space between glass beads (black) in a  $150 \times 150 \mu\text{m}^2$   
 F1:3 window at the entrance of the porous media [see Fig. 2(c)], with the  
 F1:4 suspension flowing upwards. 4 conditions of ionic strength are  
 F1:5 presented at 4 times.



F2:1 FIG. 2. Local transport and adsorption mechanisms. All scale  
 F2:2 bars are  $10 \mu\text{m}$ . (a) Time projection of confocal imaging, with the  
 F2:3 suspension flowing upwards.  $I = 10^{-6} \text{ M}$ . Steady particles  
 F2:4 appear as white dots, while moving particles are represented  
 F2:5 by their trajectories. Selected trajectories: particle 1 along wall, 2  
 F2:6 close to wall, 3 far from wall. (b) Schematic velocity profile in  
 F2:7 a pore of initial and current radii  $r_0$  and  $r$ . Dashed area shows  
 F2:8 positions favorable to adsorption:  $[r_c : r - r_p]$ . (c) Schematics  
 F2:9 of the three scales used in Figs. 1, 2 and 3(a). (d) Selected area  
 F2:10 under steady state for different flow rates  $[Q_0; 8Q_0; 16Q_0]$ , at  
 F2:11  $I = 10^{-1} \text{ M}$ . (e) Cluster formed on a surface at  $I = 10^{-1} \text{ M}$  (top),  
 F2:12 detaching and moving to a more stable position (bottom). Dashed  
 F2:13 lines highlight bead surfaces.

144 given system, while, as described below, the probability of  
 145 adsorption will significantly depend on the characteristics  
 146 and evolution of the system. Such an approach relies on the  
 147 same fundamental assumptions as the basic approach of  
 148 adsorption [31] or the ‘‘Eyring model’’ for describing the  
 149 viscosity of a simple liquid [32].

150 Then, if a particle encounters a free site, it can adsorb to it  
 151 with some probability resulting from the surface-particle  
 152 interactions and its motion characteristics. Otherwise, if it  
 153 encounters an adsorbed colloid, it will be repulsed and will  
 154 progress further along its streamline. A fundamental observa-  
 155 tion in our experiments is that no particle deposited on the  
 156 beads detaches later. Thus, neglecting in first approximation  
 157 the variation of boundary conditions due to progressive  
 158 particle adsorption, the probability of adsorption depends on  
 159 the flow rate, and is proportional to the particle concentration  
 160 and to the fraction of bead surface still available for  
 161 adsorption. Let us call  $s$  the surface coverage, i.e., the ratio  
 162 of the current number of particles adsorbed at the surface  $S$  to  
 163 the maximum possible value under our flow conditions  $S_0$ .  
 164 Then, the rate of variation of  $s$  writes as  $\partial s/\partial t = k(1 - s)$ .  
 165 In this expression  $1 - s$  is the available surface coverage,  
 166 and  $k$  is a factor including the frequency of attempt of  
 167 adsorption and the probability of adsorption for a particle  
 168 approaching a free wall. This model is a specific case  
 169 of the Langmuir approach initially developed for molecular  
 170 adsorption, but here without detachment. It solves as  
 171  $s = 1 - \exp(-kt)$ .

172 From the images, we compute the fluorescence  $F$  over  
 173 time and over a large number of beads, i.e., the whole  
 174 capillary entrance [see Fig. 2(c) and [29]]. With regards to  
 175 a saturated surface deposition  $S_0$ , for  $I = 10^{-6}$  M, the  
 176 deposition  $F/S_0$  increases gradually before reaching a  
 177 plateau value around 1 [see Fig. 3(a)] associated with a  
 178 single layer of particles (see Fig. 1,  $I_1$ - $t_4$ ). This dynamics  
 179 is well described by the above model for  $s(t)$  [see Fig. 3(a)]  
 180 with  $k = 5.0 \times 10^{-4} \text{ s}^{-1}$ , which confirms our understand-  
 181 ing of the whole process of monolayer deposition.

182 Let us now see how the process evolves when interparticle  
 183 bonding is allowed, by increasing the ionic strength. For a  
 184 relatively low ionic strength (i.e.,  $I = 10^{-4}$  M) we observe a  
 185 change in the distribution of the deposits (see Fig. 1,  $I_2$ - $t_3$ ):  
 186 the process again starts by particle adsorption on the bead  
 187 surface but it is soon followed by particle adsorption to other  
 188 particles, which leads to the formation of deposits thicker  
 189 than one particle size.

190 Under these conditions, since the particles keep the same  
 191 properties all along the process, we would expect a  
 192 continuous adsorption until filling the whole pore space  
 193 and thus clogging of the system. On the contrary, we  
 194 observe (see Fig. 1,  $I_2$ - $t_4$ ) that the deposition progressively  
 195 slows down and seems to tend to a maximum, significantly  
 196 before saturation of the whole pore space. More precisely,  
 197 the deposited amount increases at a decreasing rate and  
 198 finally tends to a plateau at long times [see Fig. 3(a)]. Thus,

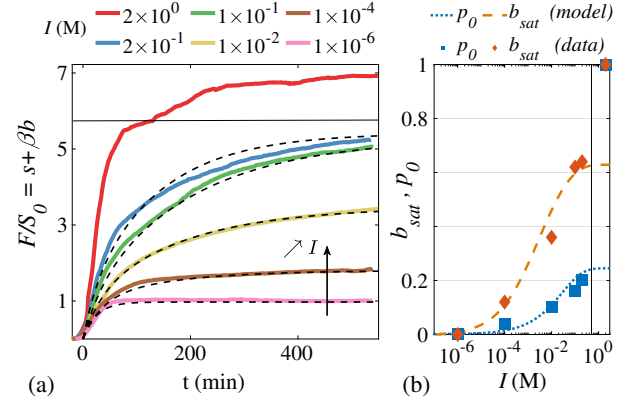


FIG. 3. (a) Overall deposition  $F$  over time rescaled by surface saturation  $S_0$ , at the entrance of the porous media for increasing ionic strengths (bottom to top). Plain lines correspond to data. Values above the horizontal line are not quantitative due to resolution limitations (see Ref. [29]). Dashed lines show the fitted model:  $s(t)$  for  $I = 10^{-6}$  M, with  $k = 5.0 \times 10^{-4} \text{ s}^{-1}$ ;  $s(t) + \beta b(t)$  for larger values of  $I$ , with  $\tau = 3.0 \times 10^4$  s. (b) Calculated and measured values for the initial bulk deposition probability  $p_0$  (dotted line, squares) and the bulk deposition at saturation  $b_{sat}$  (dashed line, diamonds), as a function of the ionic strength. The vertical line shows the limit of application of the model (i.e., suspension instability).

colloid deposition appears limited by some self-equilibrium process.

This implies that the flow evolution plays a major role: as the deposit thickness increases, the porosity decreases, and thus the local velocity increases (at constant flow rate); the drag force on the particles then increases, which decreases the probability of adsorption. Beyond some critical velocity the drag force is so large that no adsorption is possible.

This interpretation is confirmed by additional observations. By stepping the flow rate from  $Q_0$  to  $16Q_0$ , we obtain successive steady stationary deposits [see Fig. 2(d)]. The particles are washed from the large velocity regions and remain stable in the low velocity regions (i.e., around contact points), which provides a straightforward demonstration of the impact of velocity on the deposit thickness at equilibrium. At this point, it is also interesting to note that no detachment of particle is observed as long as deposition is far from its saturation value. This means that the velocity field variations do not affect the cohesion of individual particles. As we approach the stationary conditions though, during injection at a constant flow rate, some clusters of particles can suddenly detach and move slightly further in the medium [see Fig. 2(e)]. This illustrates that the local drag conditions, which initially allowed the formation of the deposit, have then evolved. The detachment of large clusters instead of individual particles likely results from the increase of the drag force on cohesive obstacles inducing a larger torque on the cluster.

Finally, with further screening of the repulsion (i.e., increasing  $I$ ), the adsorption rate is larger, and the plateau

F3:1  
 F3:2  
 F3:3  
 F3:4  
 F3:5  
 F3:6  
 F3:7  
 F3:8  
 F3:9  
 F3:10  
 F3:11  
 F3:12

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229 of deposition increases [Fig. 3(a)]. We conclude that  
 230 the particle deposition is essentially governed by some  
 231 equilibrium between the flow conditions and the colloidal  
 232 interactions. Such a qualitative conclusion matches stan-  
 233 dard theoretical analysis in that field [33], but we here have  
 234 access to a detailed description of the process, from the  
 235 local to the average level, which emphasizes the main  
 236 trends of colloidal deposition (plateaux) and provides  
 237 quantitative data for the local rate of adsorption in time.  
 238 Note that with a strong screening of the interparticle  
 239 repulsion ( $I = 2 \times 10^{-6}$  M), heavy bulk deposition occurs  
 240 at early times (Fig. 1,  $I_4-t_2$ ). Even though this coincides  
 241 with the limitation of our observation method due to  
 242 massive diffraction (see Fig. 1,  $I_4-t_3$  and [29]), particles  
 243 likely occupy a large fraction of the pore space. In that case,  
 244 no self-equilibrium prevents the clogging of the system.

245 We can describe this process through a simple model. As  
 246 a particle approaches a previously deposited particle, it  
 247 interacts with it through Van der Waals forces and electro-  
 248 static forces. This interaction is well represented by a  
 249 potential well  $\Delta\Phi$  increasing with ionic strength, as the  
 250 screening of the electrostatic repulsion increases (see  
 251 Ref. [29] for a complete description of  $\Delta\Phi$ , function of  
 252 the Hamaker constant  $A_H$ , and the particle surface potential  
 253  $\Phi_0$ ). We can thus expect that while it gets sufficiently close  
 254 to an already adsorbed particle, the former will tend to fall  
 255 in this potential well. A particle will then remain attached if  
 256 the drag force exerted by the liquid flow around it is  
 257 sufficiently small. In other words, in order to remove this  
 258 particle from its potential well, the liquid velocity around  
 259 the particle must be such that the work  $W_d = 6\pi d_p^2 \eta v$  due  
 260 to drag force during the particle displacement out of the  
 261 well (thus over a typical distance of the order of the particle  
 262 size), is larger than the depth of the well, i.e., if  $W_d > \Delta\Phi$ .  
 263 The balance of these energies defines a critical fluid  
 264 velocity  $v_c$ . For a flow through any geometry, the fluid  
 265 velocity increases with the distance from the walls, which is  
 266 confirmed by our observations (considering that, in general,  
 267 moving particles have the same velocity as the fluid) [see  
 268 Fig. 2(a)]. As a consequence, we can define a critical  
 269 position  $r_c$  at which  $v(r_c) = v_c$ , such that only the particles  
 270 situated at a distance greater than  $r_c$  from the pore axis may  
 271 adsorb [see Fig. 2(b)]. On the other side, particles are  
 272 sterically constrained at a distance (from particle center  
 273 to wall) larger than one particle radius  $r_p$ . Assuming  
 274 homogeneous particle dispersion in the fluid, the interval  
 275  $[r_c; r - r_p]$  compared to the full pore volume defines the  
 276 fraction of particles in a place suitable for adsorption, thus  
 277 proportional to a probability of adsorption  $p$ . For a given  
 278 flow rate the exact critical values and adsorption probability  
 279 depend on the detailed boundary conditions, i.e., the porous  
 280 medium structure, which changes with the volume of  
 281 deposited particles. On average (over a larger number of  
 282 pores), we thus expect a probability  $p$  depending on the  
 283 fraction of pores occupied by deposited particles, i.e.,  $b$ .

284 More precisely,  $b$  is the ratio of the number of particles  
 285 adsorbed to other particles  $B$  to the maximum number of  
 286 particles that fit in the pore space  $B_0$ , excluding the  
 287 maximum number of particles in the surface layer  $S_0$ .  
 288 Note that the ratio  $\beta = B_0/S_0$  is a bulk to surface filling  
 289 capacity ratio. Under these conditions the adsorption rate  
 290 expresses as  $\partial b/\partial t = p(b)$ . Consistently with our obser-  
 291 vations this probability will increase with the ionic strength,  
 292 and decrease when increasing the flow rate (since in  
 293 laminar conditions, the local velocity increases in the same  
 294 proportion) or, equivalently, when decreasing the porosity.  
 295 We can also define  $b_{\text{sat}}$  as the maximum value reached by  $b$   
 296 under given conditions.

297 For example, for a cylindrical pore throat of radius  
 298 initially equal to  $r_0$  but reduced to  $r$  by deposited particles  
 299 along its wall, the adsorption probability writes  $p(r) =$   
 300  $1 - r_c^2/(r - r_p)^2$  [see Fig. 2(b) and Ref. [29]]. In that case  
 301 we have  $b = 1 - r^2/r_0^2$ , from which we deduce  $p(b)$ .  
 302 Remarkably, the simple expression  $p_0(1 - b/b_{\text{sat}})$  is a  
 303 good approximation of  $p(b)$  for all pore sizes  $r$  greater  
 304 than  $r_p$ , i.e., as long as the pore is not fully clogged (see  
 305 Ref. [29] for details and approximated forms of  $p_0$  and  
 306  $b_{\text{sat}}$ ). Looking at the particle deposit as a whole, as a first  
 307 approximation, it means that the process could still globally  
 308 be considered as a simple deposition process (see above)  
 309 with regards to a saturation value, i.e.,  $b_{\text{sat}}$ , depending on  $I$ .

310 Extrapolating this result to the more complex pore  
 311 structure in a bead packing suggests writing the adsorption  
 312 rate as  $\partial b/\partial t = k's(1 - b/b_{\text{sat}})$ , where the factor  $s$  appears  
 313 as the existence of an initial surface layer is required for  
 314 bulk adsorption. Within our framework,  $k'$  is a factor  
 315 including (i) the frequency of attempt of adsorption and  
 316 (ii) the probability of adsorption of a particle approaching a  
 317 deposited particle. Therefore, we can write  $k' = p_0/\tau$  with  
 318  $\tau$  a characteristic time of adsorption attempt, considered as  
 319 constant here, while  $p_0$  strongly depends on the ionic  
 320 strength conditions.

321 From the above general expression for the bulk adsorption  
 322 rate  $\partial b/\partial t$ , and the expression for the surface deposition  $s$ ,  
 323 we can analytically solve the bulk deposition dynamic **1**  
 324 as  $(b/b_{\text{sat}}) = 1 - \exp\{(k'/k)(1/b_{\text{sat}})[1 - \exp(-kt) - kt]\}$ .  
 325 Besides, after rescaling by the surface layer fluorescence,  
 326 the total deposition  $F = S + B = sS_0 + bB_0$  writes  $F/S_0 =$   
 327  $s + \beta b$ . The predicted trends for  $F/S_0$  agree well with all our  
 328 observations [see Fig. 3(a)]: initial deposition limited by the  
 329 formation of a surface layer ( $F/S_0 = 1$ ), and further  
 330 deposition (if  $k' \neq 0$ ) decaying to the asymptotic value  
 331  $1 + \beta b_{\text{sat}}$ . This is valid up to high ionic strength values  
 332 ( $I \sim 10^0$  M), above which the particles may easily form  
 333 large loose clusters, which enhances the clogging proba-  
 334 bility. To compare our expression for  $F/S_0$  to the exper-  
 335 imental dynamics,  $\beta$  is fixed at 6.7. Comparison for  
 336  $I \in [10^{-6}; 2 \times 10^{-1}]$  M, by fitting  $k'$  (therefore  $p_0$ ) and  
 337  $b_{\text{sat}}$ , shows modeled dynamics in good agreement with  
 338 the experimental values [see Fig. 3(a)]. Independently, from

339 the model, the values of  $p_0$  and  $b_{\text{sat}}$  are calculated by slight  
 340 adjustment of  $A_H$  and  $\Phi_0$  around their theoretically predicted  
 341 values [29]. The single value  $\tau = 3.0 \times 10^4$  s then allows us  
 342 to match experimental and calculated  $p_0$  and  $b_{\text{sat}}$  over  
 343 5 decades of ionic strength [see Fig. 3(b)]. We therefore  
 344 validate our ability to predict the impact of the ionic strength  
 345 on the bulk deposition probability and saturation.

346 Overall, the competition between the interparticle inter-  
 347 actions and the evolutive hydrodynamics allows us to  
 348 completely describe the deposition state and rate. The  
 349 resulting full model can be used to compute the rate of  
 350 deposition along all regimes, from early single layer surface  
 351 adsorption to limited multilayers or clogging. The addi-  
 352 tional approximated model can be used to quickly estimate  
 353 the existence of a critical saturated state of deposition. Such  
 354 tools can significantly change the accuracy of contaminant  
 355 transport predictions. More generally, the mechanism of  
 356 controlled limited deposition can pave the way to new  
 357 filtration, cleaning, or sorting systems, with controlled  
 358 adaptive pore size distributions. As an important next step,  
 359 confronting these local mechanisms to deeper sections  
 360 **2** (same dataset) will help answer the fundamental question  
 361 of depth propagation.

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