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Factors Controlling Colloidal Transport in a Karst Aquifer

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1 introduction

It is well known that many elements are present in natural waters not only as truly dissolved entities but also as colloidal matter. It is therefore important to understand the migration behaviour of groundwater colloids to assess their role on the transport of contaminants. Colloidal transport depends on several physical and chemical phenomena (McCarthy and Zachara, 1989) involving both particle characteristics and aquifer properties. Until now, most of the research on groundwater concerned porous media and was developed on the basis of the classical advection-dispersion models (Ryan and Elimelech, 1996). Column experiments have been used to test the influence of several factors on transport behaviour, however numerous unknowns still remain, mostly concerning the chemical aspects.

In porous media, most models derive the colloid deposition from filtration theory. The pseudo-first-order rate constant used for attachment plays a major influence on the colloidal fluxes; it is a function of the collector diameter, the medium porosity, the water velocity and the single-collector efficiency, η, and the sticking efficiency, α. The η coefficient is calculated from the filtration theory by using the single collector efficiency case and appears to give reliable estimates (Johnson et al., 1996; Saiers et al., 1994a). According to the theory, bacteria, or latex spheres ($\rho = 1.05$ g/cm^3), having a size of 0.5-1.5 μm may travel at longer distances than other sizes, for most porous field conditions ($10^{-2} < u < 2.5 m/d$ and $0.1 < d_c < 10$ mm). For clays, the size range associated to long travel distance is between 0.2 and 0.8 μm, assuming a density of 2 g/cm^3.

The major role of solution chemistry and particularly of surface charge is emphasised in numerous experiments, and is generally lumped in the α parameter. For colloids in contact with collector surfaces of similar charge, the percent of recovery (R) is almost 100% and the colloids are not retarded (Saiers et al., 1994a,b). Conversely, if colloids show a charge opposite to the matrix, the deposition is close to 100% and decreases only when all the sites are occupied, (Saiers et al., 1994a; Johnson et al., 1996). In that case, the colloids are removed at a rate predicted by the η value. Several intermediate cases were described and emphasise the role of zeta potential (Tipping et al., 1993; McCaulou et al., 1995) and ionic strength (Puls and Powell, 1992; Tan et al., 1994; Fontes et al., 1991). Different authors (Kia et al., 1987; Khilar and Fogler, 1984; Litton and Olson, 1993; Cerda, 1988; Ryan and Gschwend, 1994) used Van der Waals and double layer forces to adequately predict pH and I values for colloid detachment (or attachment). However Litton and Olson (1993), Ryan and Elimelech (1996) and Nocito-Gobel and Tobiason (1996) demonstrated that even though detachment pH could be predicted, the theoretical values of α differed from measured values. This can be explained by the heterogeneous nature of matrix surfaces (Ryan and Elimelech, 1996; Loveland et al., 1996).

The numerous column experiments showed that the major colloids occurring in shallow groundwater, i.e. clays, bacteria, natural organic matter (NOM), silica, iron oxydes, can move through porous media. In natural and man-influenced conditions, the ubiquitous presence of NOM, and in some cases of PO_4^{2-}, involves a very low value for α (0.001 to 0.01). This explains the occurrence and indications of movements of colloids in several natural media (Penrose et al., 1990; Gschwend and Reynolds, 1987). Artificial colloids such as latex beads are generally submitted to much higher deposition rates (Harvey et al., 1989). The few colloidal, namely bacteria, transport experiments done in the field over several meters showed important colloid straining but

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the portion of colloids arriving at the monitoring point presented only slightly skewed breakthrough curves and no or minor retardation (Harvey et al., 1993) compared to solutes. Hence, the bacteria arriving at the peak were not submitted to deposition.

This paper presents results from a karst aquifer. The main specificity of karst is the existence of large conduits, inducing water velocities orders of magnitude higher than in porous media. The filtration theory is not relevant under these conditions but chemical factors can influence colloidal transport by the possibility of aggregation or sticking to wall conduits. This is what we intend to analyse in this paper. Due to the diameter of conduits, the water velocity and the length scale involved, laboratory experiments are not feasible with a correct scaling. This is why this study is based on field data from a karst aquifer chosen for its interesting colloidal content.

2 site

The 60 km^2 Noiraigue basin is located in the Swiss Jura mountains at an altitude ranging from 1000 to 1350m. Average rainfall, over the last 70 years, was about 1500 mm/y. One third of the catchment area is represented by the flat 'Vallée des Ponts' covered by peat bogs and drained by the 'Bied brook' flowing in a major sinkhole, and two third by the surrounding hills made by calcareous anticlines. The surrounding anticlines are drained very efficiently by the epikarst revealed by numerous sinkholes. Therefore no surface flow occurs in this part whatever the rainfall intensity is. The main aquifer is developed in 400 m of folded Jurassic limestone. The existence of a 300 m deep layer of Molasse in the central valley suggests that the karst aquifer underlying this part must be saturated. The karstic network is well developed with water velocities varying typically from 10 to 60 m/h and discharge at the Noiraigue spring, unique basin outlet, ranging from 0.5 to 20 m^3/s. The site is equipped for continuous measurements of water level, temperature, electric conductivity, turbidity, and rainfall on the Bied brook and at the Noiraigue spring. Samples were taken almost weekly from May 94 to November 95 and at shorter time intervals for specific events.

3 methods

Several methods, described in detail elsewhere (Atteia and Kozel, 1997; Atteia et al., 1998), were used to determine the size and composition of particles found in the geological layers and in the sampled waters. Briefly, the particle size distributions were determined by laser sizer (Galai CIS), based on single particle counting. The mineralogical composition of the main geological formations were determined by X-ray diffraction (Scintag XRD 2000). For water samples, one litre of suspension was centrifuged at 1800 g during 20 min, and the pellet was deposited directly on glass plates for XRD analysis. For some samples, 10 l of suspension were first fractionated by continuous ultracentrifugation, to obtain several size classes, prior to XRD analysis.

During the first campaigns, samples were filtered using a home-made cascade filtration stack which allowed fairly accurate size cutting and very limited clogging (Atteia et al., 1998). The samples were divided into five fractions from larger than 2 μm to smaller than 0.05 μm, and the Fe content was analysed by AAS (Perkin Elmer 5100 PC). For several samples, the concentrations in suspensions and retained onto filters were analysed in order to calculate the mass balance. The difference between raw sample and the sum of the fractions, was below 10%. However pumping and long filtration time could enhance Fe precipitation or growing of existing Fe particles. The $< 0.05\mu$m Fe fraction might therefore be the only relevant one.

Bied and Noiraigue waters of four campaigns, filtered on Nuclepore polycarbonate filters (0.8 and 2.0 μm), were analysed in detail by electron microscopy. The analysis of individual particles was processed with a SEM-EDS (Link Analysis Si(Li) detector on a Jeol JSM-35 microscope). Approximately 1000 particles were randomly chosen and analysed under identical conditions. The results are given in intensities of the X-ray emission of each element. This was complemented by TEM-EDS on the same samples in order to analyse smaller particles; in that case, only particle types, and not intensities, were recorded.

Pyrolysis-GC-MS has been used to characterise the composition of natural organic matter (NOM). Three sampling campaigns were done, camp.1 and 3 (12.95 and 2.96) were done at low flow conditions, and camp.2 at medium flow. All the used analytical conditions, and calculations were same as Bruchet (1990) ones. The results are given as concentrations of the species detected by GC-MS after the pyrolysis step, which might differ from the original molecules composition. Due to this pyrolysis effect and the varying pyrolysis efficiencies according to the organic matter composition, the results can be affected by the NOM type. However, if the samples come from the same environment, pyrolysis-GC-MS produces highly informative comparative results.

Two tracing experiments were performed, one at low, and the other at medium flow conditions. Tracers were injected from six sinkholes located at the border of the valley, the Noiraigue spring being continuously monitored for breakthrough curve. Three different dyes (Fluorescein, Duasin and SulforhodaminG) and four bacteriophages (H40, H6, H4, T7) were injected within 2 hours. The bacteriophages are phages cultivated on marine bacteria populations. After destruction of the bacteria phages are thus unable to reproduce under freshwater conditions. They are counted, down to one phage/ml by cultivation techniques on bacterial gels. Several tests

Table 1. Average concentrations of solutes in waters from the basin, TOC: Total organic carbon, FeT: total iron, FeF: filtered iron, V: average from 6 points over the valley, Bh: Borehole, Noi: Noiraigue spring, , *: Estimated from: 30% Valley + 70% Borehole

	pH	TOC mg/l	Ca mg/l	Mg mg/l	Na mg/l	K mg/l	Si mg/l	Mn μg/l	FeF μg/l	FeT μg/l	PO_4 μg/l	HCO_3^- mg/l	SO_4^{2-} mg/l	NO_3^- mg/l	Cl^- mg/l
V	7.45	16.2	87.7	3.6	2.[illegible]	3	1.6	51.3	248	615	106	275	5.7	6.8	5.3
Bh	7.79	1.1	73.9	13.3	3.9	-	1.5	0	8	28	79	-	7.3	6.1	8.5
Noi	7.39	3.4	81.5	4.6	2.4	1	1.4	0	22	58	89	261	5.4	7.7	5.4
Noi*	7.69	5.6	78	10.4	3.4		1.5	15.4	80	204	87		6.8	6.3	7.5

done in rivers showed their potential use as tracers (Rossi et al., 1997).

4 results

The composition of surface waters from the valley, from a borehole and from the Noiraigue spring are compared in table 1. The borehole is located at one border of the valley, the pumped water coming only from the hill part of the basin. The concentrations of SO_4^{2-}, NO_3^-, PO_4^{3-}, and Si are quite similar in the three media. Cl^-, Na^+ and Mg^{2+} are enriched in the borehole waters, whereas Ca $^{2+}$ content is higher in the valley waters rather than borehole or spring. Total organic carbon (TOC), Fe and Mn content are very low in borehole and spring as compared to the valley.

A rough estimate of the Noiraigue spring composition can be obtained from the composition of the valley and borehole waters multiplied by the area coverage of each type of media. The area of the valley, estimated from basin delimitation, represents *ca.* 30% of the total area. The estimated composition is in the same range as the measured one for most of the elements, except for Fe and Mn, and to a minor extent for TOC.

The different types of particles were described in a previous paper (Atteia et al., 1998) in relation with their potential origin. The particle content vary between 0.5 and 10 mg/l whereas the solute one is close to 200 mg/l. The main types of natural particles are: grain minerals (calcite, quartz, feldspars) from geological deposits, clays, which originate from quaternary deposits and limestone, organic matter coming from peat bogs, and specific Fe-Ca particles. These Fe-Ca particles seem to be formed during the oxidation of globular organic matter when these ones enter the drainage network or the Bied brook (Couture et al., 1998). We will focus here on the evolution of the composition of particulate matter from the Bied brook to the Noiraigue spring in order to infer some transport properties of the identified particles.

The average mineralogical composition of the geological layers is given in table 2. The XRD analyses revealed some differences in the composition of the grain mineral fraction of the suspensions. With reference to quartz, the Bied brook contains 40% more feldspars than Noiraigue but only a third of the content of calcite and dolomite of Noiraigue waters. Concerning clays, the composition of the suspensions from both points were statistically similar, made of *ca.* 50% of mica, 25% of kaolinite and 25% of chlorite.

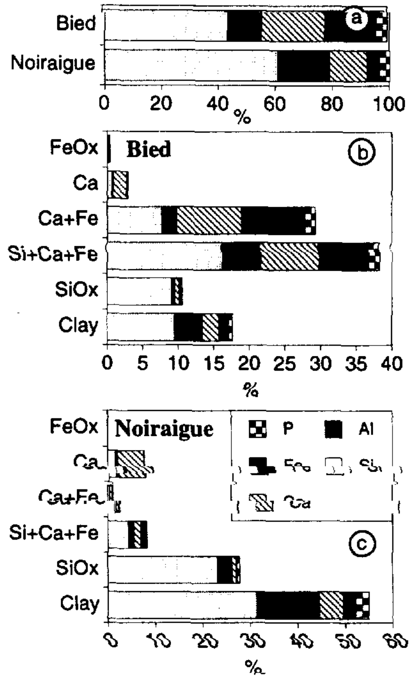

Fig. 3. Proportion and composition of particles on Nuclepore filters differentiated according to their type determined by major element content, from EDS measurements

The analysis of the SEM-EDS results required a classification of the particles in approximate types. This was achieved by determining the major element in each particle by using the ratio of intensity of one element to the sum of the intensities (Si+Al+Ca+Fe+P+Mn). Most of the particles included Si and Al in similar ratios (*ca.* 2.5:1 in intensities). As a large proportions of minerals were identified as clays by XRD, we considered that the particles containing 60% of a Si+Al fraction were clays. The second group was Si oxides particles, containing more than 70% of Si. The third was called 'Ca+Fe par-

Table 2. Estimates of mineralogic content of geological layers: -: absent, +: rare, ++: common, +++: abundant, ++++: very abundant; Quat.: quaternary, in these marls (smectites) is for specific inter-layered smectites containing high amounts of Al and Fe

deposit	Mica	Smectites	Kaolinite	Chlorite	Dolomite	K-Feldspars	Plagioclase
Quat. Marls	+++	(+++)	+++	++	+	++	++
Molasse	++++	+++	-	++++	++	++	++++
Cretaceous	++	+++	++	+	-	+	-
Jurassic	++++	++	++	+	+++	+	-

ticles' if the Ca+Fe fraction represented more than 50% of the total. The fourth fraction consisted of particles having an Si+Ca+Fe fraction higher than 70%. The last fraction were calcium particles and iron oxides, in which either Ca or Fe represented more than a half of total intensity. Figure 1b,c shows the relative abundance of each particle type and the composition of these fractions. The composition of the clay, Si oxide and Ca particles and their relative amount are very similar in both media. The main difference arises from the Si+Ca+Fe and Ca+Fe particles which compose 67% of the total particles in the Bied brook, but less than 10% in the Noiraigue suspensions. As these particles are the main source of particulate Fe, this justifies the sharp decrease of particulate Fe content from 18 in the Bied to 4% at Noiraigue (fig. 1a). The TEM-EDS analysis revealed the same patterns, and showed that most of the Ca+Fe particles were in the smaller fraction, *i.e.* below 0.5 μm (table 3).

The speciation of particulate Fe by filtration, acid extraction and wet analysis also showed the same kind of results (fig. 2). A decrease of an order of magnitude in particulate Fe content between the Bied brook and the Noiraigue spring was found. This approach also revealed that in most Bied suspensions samples, a half of the iron was included in the fraction below 0.05 μm. Beside the decrease in the particulate Fe, this 'soluble' Fe also decreased, of almost an order of magnitude. Globally the Fe content of the Noiraigue suspensions is lower than 10% of the Bied one.

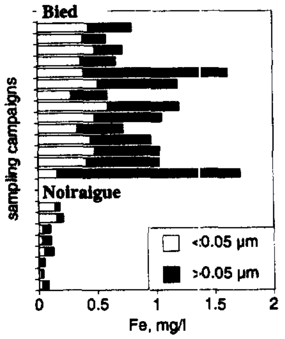

Fig. 2. Proportion of 'soluble' and 'particulate' Fe fractions obtained by cascade filtration

Table 3. Percentage of particles by size fractions for Bied brook and Noiraigue spring suspensions. Results obtained by TEM for size and TEM-EDS for particle composition

	< 0.5 μm	0.5-1 μm	> 1 μm
Bied			
FeOx	71	25	4
Fe/Ca	82	16	2
Clay	34	58	8
Noiraigue			
Si/Ca/Fe	47	45	8
Fe/Ca	85	15	0
Clay	36	50	14

Natural organic matter (NOM) is difficult to characterise and analytical techniques often give only qualitative results. Pyrolysis-GC-MS allows to make a broad classification of the major NOM components, by splitting them in four classes. The results of three sampling campaigns are given in fig. 3. The peat polysaccharides (PS) of the first campaign contain significant amounts of 1-hydroxy-2-propanone, which is a typical pyrolysis product of dextran, a starch-like substance. Starch is fairly easily degradable, and so the presence of these fragments usually indicates that this portion of the organic matter is fresh. In the second and third campaigns, the largest contributor to the PS group is furaldehyde, beside 1-hydroxy-2-propanone. Furaldehyde is a typical pyrolysis product of cellulose. The other samples have no significant amounts of hydroxypropanone in their pyrolysates. Amino sugars (AS) come from the degradation of the cell walls of microorganisms. In the Polyhydroxyaromatics (PHA's) fraction, benzene and phenol are always the major constituents with phenol often representing more than 2/3 of this fraction. The main variation in NOM composition is a clear decrease of PS content compensated almost only by an increase of PHA along the water pathway. The two other fractions do not show very distinctive evolution. This behaviour is well marked at low flow but much less clear at medium flow.

The Fe and TOC content of samples taken at short intervals during a high flow event are depicted in fig. 4, in relation with the evolution of discharge and turbidity. The completely different behaviour of organic matter and Fe is clearly evidenced. The Fe content presents a sharp peak at the very beginning of the discharge

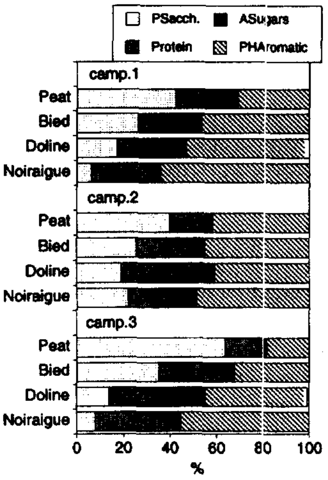

Fig. 3. Proportion of NOM constituents obtained by Pyrolysis-GC-MS for three campaigns on several points of the basin. Doline is the major valley sinkhole

increase, when the turbidity is the highest. A much smaller peak appears when TOC increases. This fast Fe peak had already been observed at the same spring by Bouyer (1997) in the 70's. At contrary, the organic matter content increases only two days later, the peak being much less marked.

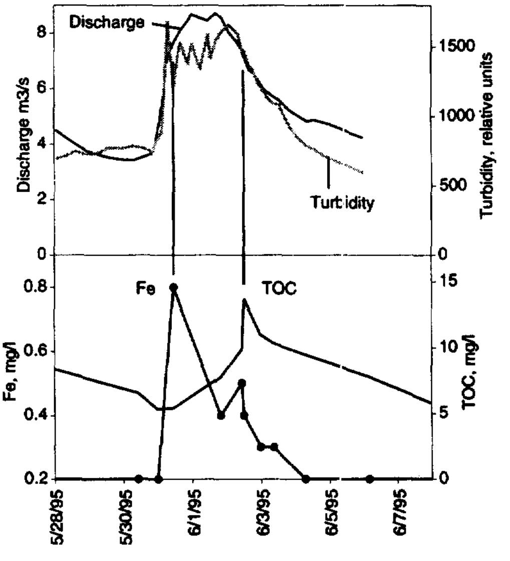

Fig. 4. Evolution of total Fe and TOC content during a high flow event at the Noiraigue spring, related to discharge and turbidity

The general results of the tracers tests are described elsewhere (Atteia et al., 1996), we focus here only on the differences observed between the arrival of a dye, Duasin, and a small colloidal tracer (50-100 nm), a bacteriophage. The main differences between these two tracers is the percent of recovery, close to 3 % for the phage, compared to 90 % for the soluble tracer. However the arrival time are very similar (fig. 5). In order to compare in detail the two curves, a simple model of transport with dispersion has been fitted, the parameters being displayed on fig. 5. It appears that the phage presents a significantly lower dispersivity.

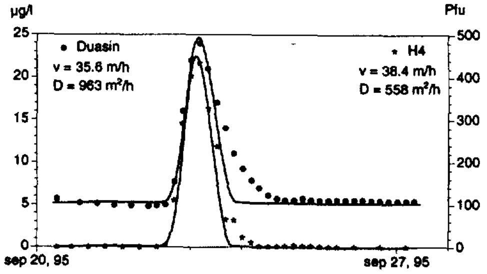

Fig. 5. Tracing experiment on a 2.4 km distance in karst, v, velocity, and D, dispersivity values were obtained by fitting, for Duasin and bacteriophage H4 respectively, pfu: phage forming units

5 discussion

The analysis of transport data directly from field experiments is a complex task as many parameters often influence the results simultaneously. We will therefore use some assumptions to analyse the field results given above. We first assume that two major components enter the aquifer: water from the valley, characterised by a high colloidal and NOM content, and the one from the surrounding anticlines, poor in NOM and colloids. The composition of the valley waters is fairly well known, but water from the anticlines should vary in space and we have only one sampling point. The second assumption is that the amount of water arriving to the spring is a mixture of both waters in proportions equal to the area of each domain, *i.e.* 30 % for the valley and 70 % for the anticlines. This assumption might be acceptable on a long time scale but some variations occur in the short term due to different travel times and stocks of each domain.

The first important result is that some types of colloidal particle can be transported through the karst aquifer at a velocity similar to solute one, although a large part of the colloids seems to be retained. This is demonstrated by the bacteriophage tracing experiments. This result can be compared to Harvey's experiments (Harvey et al., 1989), the transport length scale being two orders of magnitudes higher.

Concerning clays, it is difficult to state about their transport velocities as they are ubiquitous. However the presence of chlorite at the Noiraigue spring, almost absent from the aquifer rocks, suggests that clays can travel through the whole aquifer.

NOM is also able to be transported through the whole aquifer; however the conservative aspect of this transport is difficult to state. In fact, fig. 4 clearly shows a strong increase of NOM content at the end of the high flow event, 2 days after the peak beginning. A 2 days lag is also the shortest lag obtained from tracer experiments from the main valley sinkhole to the Noiraigue spring, at high flow. This NOM peak should then correspond to the arrival of a large proportion of water from the valley. We calculated an average TOC of the Noiraigue waters (5.6 mg/l) higher than the measured one (3.4 mg/l). This might be due either to a non-conservative behaviour or to an over-sampling of low flow conditions, compared to a poor sampling of NOM rich high flow waters. More sampling of high flow events is needed to determine if any NOM is retained in the aquifer.

The NOM composition seems to evolve along the whole water pathway, *i.e.* from the peat to the Bied brook and from the main sinkhole to the spring. This evolution is characterised by a reduction of polysaccharides and an increase of polyaromatic proportions within the large molecules broken by pyrolysis. This increase of the degree of aromaticity of humic and fulvic acids is a classical indices of NOM maturation in the environment. The evolution from peat to Bied brook might be related to the major change in chemical conditions, whereas from the Bied brook to the main sinkhole, mixing phenomena occur. The addition of simple PS from river algae is excluded due to winter sampling. The important difference between the main sinkhole and the spring cannot be related to the small differences in water chemistry but might be a maturation of the organic matter during the aquifer transit time. This maturation is fairly quick as it happens in 10 to 15 days, the residence time at low flow conditions (Atteia et al., 1996). However the hypothesis of a maturation seems plausible as it is almost absent at medium flow, when the travel time is shorter that 7 days.

The most striking feature concerning the composition of the particulate matter is the quasi-absence of Ca-Fe particles in the Noiraigue spring, whereas these constitute the major particulate fraction in the Bied brook. This has also been confirmed by TEM-EDS measurements. Beside, the 'dissolved' (< 0.05 μm) Fe is also retained in the aquifer, decreasing by an order of magnitude from Bied to Noiraigue. The almost unique source of Fe and NOM is the valley; these two components are therefore injected in the aquifer jointly. NOM seems to follow the water from the valley, its concentration being related to the proportion of this water to the total discharge at the spring. As, in average, the ratio Fe/NOM is more than twice smaller at Noiraigue than in the Bied, a major part of the iron is accumulated in the aquifer.

A peak of Fe clearly occur during the first rise of discharge, when only water coming from the deep part of the aquifer is flowing. This Fe does not come from the water of the current flood but from previously deposited Fe, resuspended during the first rise of the hydrograph. This deposition can be due to (i) simple sedimentation, (ii) aggregation followed by sedimentation, or (iii) sticking to the conduit walls. The small size (0.2 to 0.5 μm for most of them) of Fe-Ca particles suggests that they cannot totally settle, because much larger particles, of some μm, are always present in Noiraigue waters, even at low flow. It might be difficult to differentiate between the two other phenomena indicated above, but they undoubtedly involve a chemical aspect. Therefore it seems that the specific nature of Fe-Ca particles enhances their sticking ability, whether between them or with conduit walls. The Fe oxyhydroxide particles are known for their high reactivity in the environment (Davison and Vitre, 1992), a similar reactivity could be attributed to Fe-Ca particles. In fact, these particles are mostly formed during the water flow from the peat to the Bied brook (Couture et al., 1998). Recently formed particles generally present a high reactivity, which could explain the specific behaviour of Fe-Ca particles in the studied karst aquifer.

6 conclusion

A skillful analysis of particle composition in relation with hydrodynamics showed that colloids can be transported over several kilometres in a karst aquifer. When colloidal transport occurs, the colloid velocities are similar to the one of solute tracers, their dispersivity being smaller. The amount of particle retained seems to differ according to the type of particle considered. This is fairly similar to experiments in porous media, except the scale of transport, which is two or three orders of magnitude longer.

The strong effect of particle composition on their transport properties, described for porous material, exists in karst too. Although more high flow events sampling is necessary, the Fe-Ca particles seem to be deposited in the aquifer due to their specific chemical composition. The reactivity of the particles might play a crucial role in the two main deposition processes in karst: aggregation followed by sedimentation or sticking to conduit walls. When subjected to these processes, particles will be highly retarded and might appear at the spring only during high flow events, as a consequence of resuspension. This also suggests that a strong evolution of such particles is possible during this long retention time in aquifer sediments. Therefore, the study of the potential role of colloids as contaminant carriers might take into consideration both hydrodynamics and particle chemistry. A change in particle composition or particle-contaminant chemical bonds, able to occur within the karstic sediments, might be considered.

6.0.1 acknowledgements

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