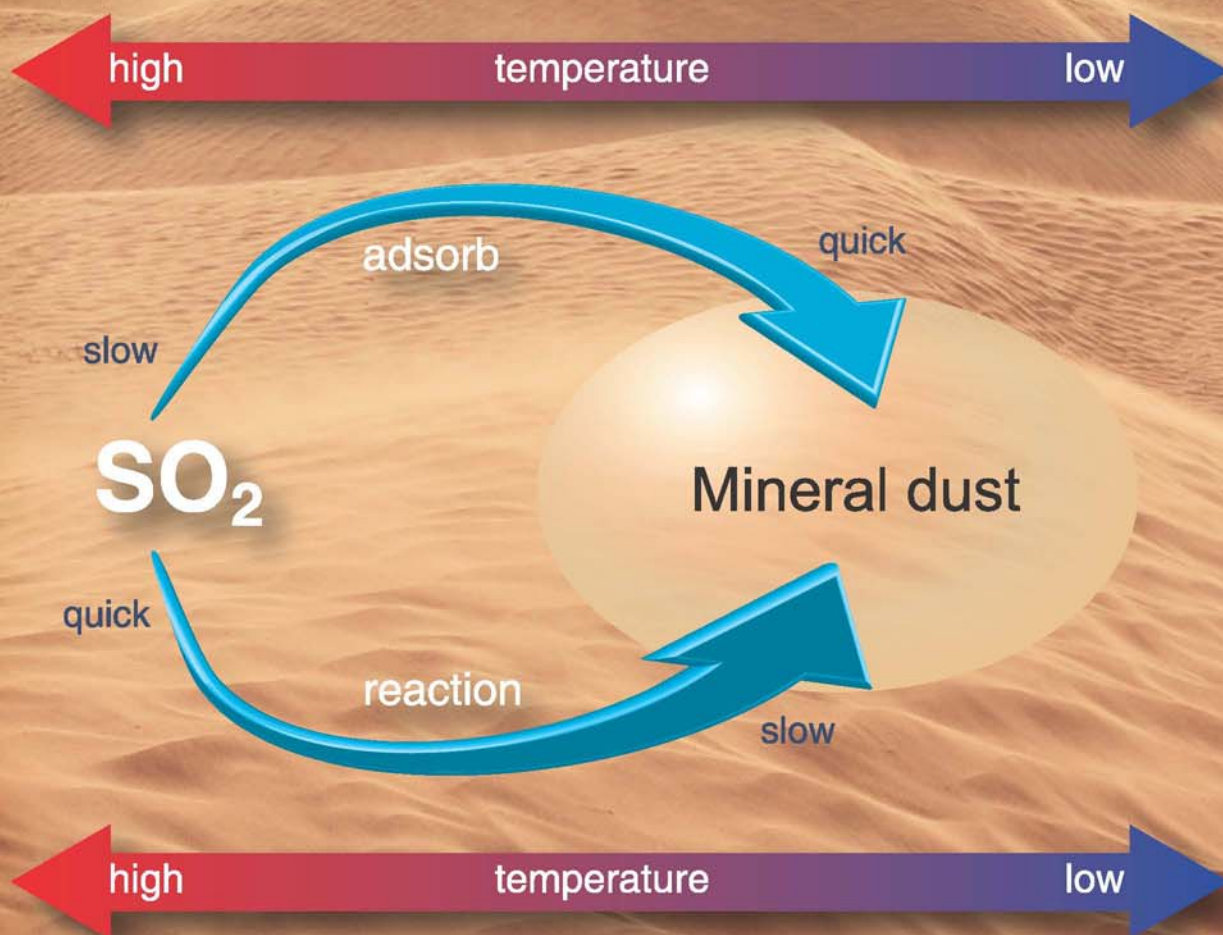


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# Humic acid transport in saturated porous media: Influence of flow velocity and influent concentration

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## ABSTRACT

Understanding the transport of humic acids (HAs) in porous media can provide important and practical evidence needed for accurate prediction of organic/inorganic contaminant transport in different environmental media and interfaces. A series of column transport experiments was conducted to evaluate the transport of HA in different porous media at different flow velocities and influent HA concentrations. Low flow velocity and influent concentration were found to favor the adsorption and deposition of HA onto sand grains packed into columns and to give higher equilibrium distribution coefficients and deposition rate coefficients, which resulted in an increased fraction of HA being retained in columns. Consequently, retardation factors were increased and the transport of HA through the columns was delayed. These results suggest that the transport of HA in porous media is primarily controlled by the attachment of HA to the solid matrix. Accordingly, this attachment should be considered in studies of HA behavior in porous media.

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## Introduction

The presence of humic substances in natural waters can cause various environmental and health problems. Humic acids (HAs) are the dominant type of aqueous organic molecules in most surface water and near-surface groundwater systems, and their behavior can significantly enhance or delay contaminant transport. HAs have a high affinity for aqueous metal cations; therefore, the fate of these metals can be controlled by the behavior of HA (Pandey et al., 2003; Ge et al., 2007; Wang et al., 2009; Zhang et al., 2009; Brigante et al., 2010; Chang Chien et al., 2010; Cao et al., 2010). The presence of HAs has been found to enhance the aqueous solubility of organic pollutants and to facilitate transport of such pollutants through soils (Williams et al., 2000) and other porous and fracture systems (Murphy et al., 1990; Ding and Wu, 1995; Murphy and Zachara, 1995; Haberhauer et al., 2002; Franchi and O' Melia, 2003). Soluble HAs have also been shown to increase the retention of organic pollutants (Conte et al., 2001), which in turn reduces the mobility of these

pollutants. Moreover, through their interaction with actinide ions such as the uranyl ion (Cherwinski et al., 1994), HAs may exert a crucial influence on the mobility of uranium (Artinger et al., 1998, 2002). However, whether HA enhances or retards the transport of pollutants depends on the solid matrix of the media and the solution properties. Therefore, HA mediated migration is important for the risk assessment of contaminant environmental behavior, and the accurate modeling of contaminant transport relies on the ability to describe the transport properties of HAs.

HAs comprise a subclass of humic substances that generally display macromolecular and colloidal characteristics. HAs contain both hydrophobic and hydrophilic moieties as well as many functional groups such as carboxylic, phenolic, carbonyl and hydroxyl groups connected to aliphatic or aromatic carbons (Hering and Morel, 1988). The existence of such carboxylic and phenolic groups results in HA being predominantly negatively charged in aqueous solutions (Cornel et al., 1986). These unique properties affect the transport of HA in different environmental media. Accordingly,

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understanding the fate of HA in the environment, especially its transport in porous media, can provide important and practical evidence for accurate prediction of organic/inorganic contaminant transport in different environmental media and interfaces. Among the different environmental media and interfaces, chemical transport in porous media is very important and has long been the focus of studies conducted by hydrologists and soil scientists due to its crucial applications in civil and agricultural engineering.

Most studies of the environmental behavior of HA that have been conducted to date have focused on the adsorption of HA onto solid surfaces under different conditions (Vermeer et al., 1998; Avena and Koopal, 1999; Specht et al., 2000; Pehlivan and Arslan, 2006; Moura et al., 2007; Wang et al., 2010). However, the transport of HA in a water-filled porous solid system and its governing mechanisms are still not well understood. The factors controlling the transport of HA in porous media may be classified into two types: (1) mechanical factors such as molecular shape, flow rate and HA concentration; and (2) physicochemical factors, such as ionic strength, pH and porous media surfaces. These factors are interrelated since molecular shape is primarily dependent on the solution pH and ionic strength. Weng et al. (2002) evaluated the transport of HA in a copper contaminated acid sandy soil and found that its breakthrough curves were characterized by a rapid, relatively sharp front followed by a plateau at a lower HA concentration than in the influent solution. Wei and Shao (2007) found that the transport of HA through soil columns varied greatly with soil types and the transport was mainly influenced by clay content and cation exchange capacity, which enhanced the adsorption of humic acid, and thus, retarded the transport in soils. Wei et al. (2010) studied the HA transport through different porous media at different solution pH and ionic strength conditions. They found that decreasing pH and increasing ionic strength increased adsorption, and therefore, delayed the transport of HA in porous media. However, few studies have been conducted to evaluate the effects of mechanical factors on transport of HA in porous media, which are essential for understanding HA behaviors in porous media and for controlling pollution related with HA transport. Therefore, this study was conducted to investigate the transport of HA in different porous media, and to discuss the effects of flow rate and influent HA concentration on the transport behavior. Our objective is to understand how HA transport responds to mechanical factors of different porous media.

## 1. Materials and methods

HA was prepared from a commercial humic substance (China Medicine Group, Shanghai Chemical Reagent Corporation, China) and then purified by mixing with deionized water, after which

the pH was adjusted to 10 with 0.1 mol/L NaOH. After centrifugation, the supernatant was removed and precipitated by acidification to pH 1.5 with concentrated HCl. Next, HA was dissolved and precipitated three times, followed by dialysis to reduce the salt content. The external water was replaced in the first 5 hr and then every 12 hr until no chloride was detected. Finally, the product was collected by vacuum filtration and oven dried at 70 to 80°C for 50 hr (Wei et al., 2010).

Glass columns with a 1.9-cm inner diameter and 11.0-cm length were uniformly packed with quartz sand (grain diameters ranging from 0.5 to 1.0 mm) and river sands (grain diameters ranging from 0.5 to 1.0 mm or <0.5 mm). The average grain densities were 2.43, 2.54 and 2.52 g/cm<sup>3</sup> for quartz sand, 0.5–1.0 mm river sand and <0.5 mm river sand. The experimental design and conditions are shown in Table 1. A previous study showed that the surface tension of HA is larger at pH 8 than at lower pH values (Yates and von Wandruszka, 1999), and the fluorescence intensity is larger at pH 8 than at lower or higher pH values (Chen and Kenny, 2007). These results suggest that low pH enhances the aggregation of HA in solution. Thus, a solution pH of 8 was chosen to conduct the experiments. Prior to use, the sand was thoroughly cleaned by washing with 0.1 mol/L Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub> for 2 hr to remove surface metallic compounds and then with 5% H<sub>2</sub>O<sub>2</sub> solutions for 3 hr to remove organic impurities, followed by washing with 12 mol/L HCl overnight and then with deionized water for 5 hr (Wei et al., 2010).

The experimental setup used for the transport experiments is illustrated by Wei et al. (2010). Standard gravimetric methods were used to determine the column packing density. All column experiments were performed in duplicate. Prior to injection of the HA solution, a HA-free KCl solution that had been adjusted to the desired ionic strength and pH was introduced to the top of the column. At least 50 pore volumes of this solution were then applied to thoroughly flush the column and stabilize the pH of the system. The pore volumes were calculated as the ratio of the volume of solution leached through the column in any given time interval and the total volume of solution present inside the column at the beginning of the transport experiment (Shukla, 2013). The flow velocity of the solution was controlled by adjusting the inflow water head. After equilibrium of the column, the KCl solution was terminated, and the HA solution was injected into the column at a constant rate. At the column outlet, the effluent suspension was collected at regular time intervals and analyzed for HA concentration by light

**Table 1 – Experimental design.**

| Factor                 | Solid matrix          | pH | Ionic strength (mol/L KCl) | Flow velocity (cm <sup>3</sup> /min) | Influent concentration (g/kg) |
|------------------------|-----------------------|----|----------------------------|--------------------------------------|-------------------------------|
| Flow velocity          | Quartz sand           | 8  | 0.001                      | 1.12–5.68                            | 5                             |
|                        | 0.5–1.0 mm river sand | 8  | 0.001                      | 0.86–6.43                            | 5                             |
|                        | <0.5 mm river sand    | 8  | 0.001                      | 1.33–7.36                            | 5                             |
| Influent concentration | Quartz sand           | 8  | 0.001                      | 4.73                                 | 10                            |
|                        |                       | 8  | 0.001                      | 4.73                                 | 2                             |
|                        | 0.5–1.0 mm river sand | 8  | 0.001                      | 2.26                                 | 10                            |
|                        |                       | 8  | 0.001                      | 2.26                                 | 2                             |
|                        | <0.5 mm river sand    | 8  | 0.001                      | 3.16                                 | 10                            |
|                        |                       | 8  | 0.001                      | 3.16                                 | 2                             |

absorbance at 254 nm using a UV-VIS 8500 spectrophotometer (Techcomp, Shanghai, China). All transport experiments were conducted at  $19 \pm 1^\circ\text{C}$ .

## 2. Mathematical modeling

The one-dimensional HA transport in homogeneous and saturated porous media while accounting for HA adsorption is governed by the following partial differential equation:

$$R \frac{\partial C(x, t)}{\partial t} + \frac{\rho}{\theta} \frac{\partial C^*(x, t)}{\partial t} = D \frac{\partial^2 C(x, t)}{\partial x^2} - U \frac{\partial C(x, t)}{\partial x} \quad (1)$$

where,  $C$  ( $\mu\text{g}/\text{cm}^3$ ) is the liquid-phase HA concentration,  $C^*$  ( $\mu\text{g}/\text{g}$ ) is the mass of HA adsorbed onto the solid matrix,  $D$  ( $\text{cm}^2/\text{min}$ ) is the dispersion coefficient,  $U$  ( $\text{cm}/\text{min}$ ) is the average interstitial flow velocity,  $\rho$  ( $\text{g}/\text{cm}^3$ ) is the bulk density of the solid matrix,  $\theta$  ( $\text{cm}^3/\text{cm}^3$ ) is the porosity of the solid media and  $t$  (min) is the time.

Eq. (1) can be re-written as:

$$R \frac{\partial C(x, t)}{\partial t} = D \frac{\partial^2 C(x, t)}{\partial x^2} - U \frac{\partial C(x, t)}{\partial x} \quad (2)$$

where,  $R$  is the retardation factor.

The expression describing the linear adsorption of HA and the initial and boundary conditions for Eq. (1) are provided in our previous article (Wei et al., 2010).

In this study, CXTFIT.2.1, a computer program developed by the U.S. Department of Agriculture for estimating solute transport parameters using a nonlinear least squares parameter optimization method, was used to fit the observed data and estimate the dispersion coefficient ( $D$ ) and retardation factor ( $R$ ) at various initial concentrations and flow rates. This software has been widely used to assess solute transport parameters of porous media. The equilibrium distribution coefficient ( $K$ ) ( $\text{cm}^3/\text{g}$ ) was calculated according to Wei et al. (2010).

The fraction of HA retained in the soil column is calculated with filtration theory (Yao et al., 1971; Logan et al., 1995):

$$F_r = 1 - \frac{C}{C_0} \quad (3)$$

where,  $F_r$  is the fraction of HA retained in the column,  $C/C_0$  is the initial removal efficiency, with  $C$  and  $C_0$  being the column effluent and influent HA concentrations, respectively.

**Table 2 – Effects of solid matrix on various parameters.**

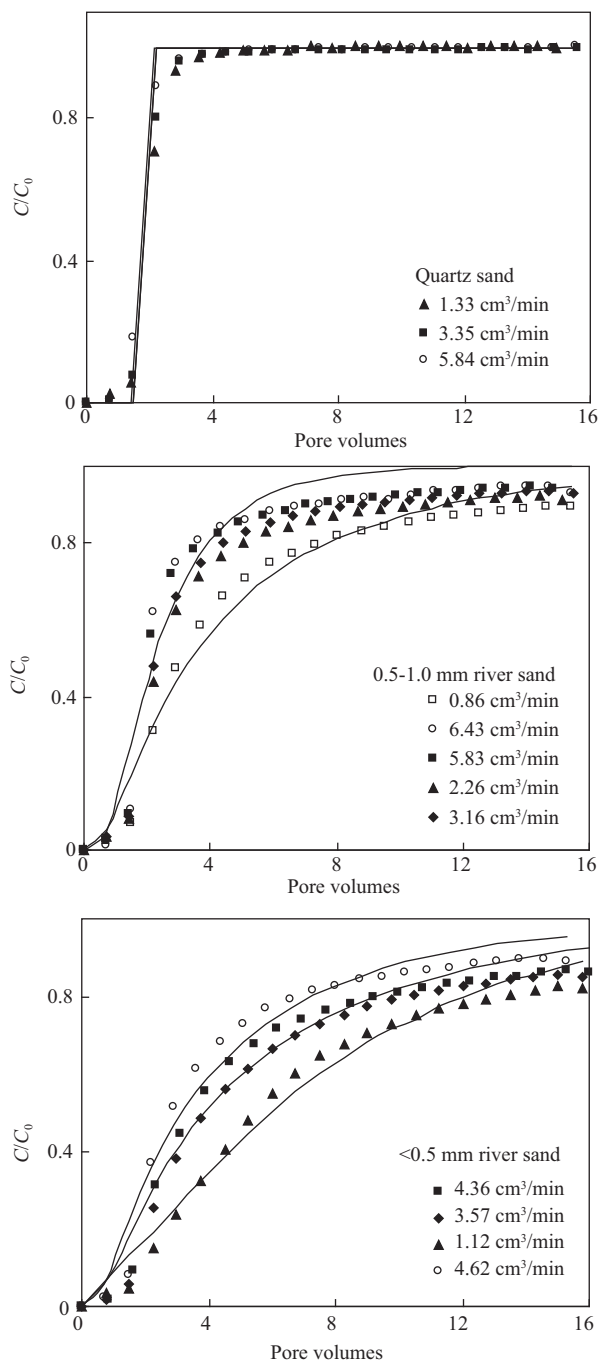
|                    | $F_r$ | $K_d$ ( $\text{hr}^{-1}$ ) | $R$  | $K$ ( $\text{cm}^3/\text{g}$ ) |
|--------------------|-------|----------------------------|------|--------------------------------|
| Quartz sand        | 0.048 | 0.89                       | 1.72 | 0.18                           |
| 0.5–1 river sand   | 0.100 | 1.79                       | 2.21 | 0.29                           |
| <0.5 mm river sand | 0.143 | 2.95                       | 2.92 | 0.44                           |

$F_r$ : the fraction of HA retained in the column;  $K_d$ : the deposition rate coefficient;  $R$ : the retardation factor;  $K$ : the equilibrium distribution coefficient.

The deposition rate coefficient ( $K_d$ ) ( $\text{hr}^{-1}$ ) of HA in the column is expressed as (Kretzschmar et al., 1997):

$$K_d = -\frac{1}{t_p} \ln \left( \frac{M_{\text{eff}}}{M_{\text{in}}} \right) \quad (4)$$

where,  $t_p = L/v_p$  is the average travel time of the HA through the column (hr),  $L$  (m) is the column length,  $v_p$  (m/hr) is the



**Fig. 1 – Breakthrough curves of HA in the quartz sand column, 0.5–1.0 mm river sand column and <0.5 mm river sand column at different flow velocities. The lines are the fitted results. The solution pH and ionic strength were 8 and 0.001 mol/L, respectively, for all samples.**

average travel velocity of HA solution,  $M_{\text{eff}}$  (g) is the total HA that came out of the column and  $M_{\text{in}}$  (g) is the total HA that was put into the column. The term  $M_{\text{eff}}/M_{\text{in}}$  corresponds to the fraction of HA recovered at the column outlet after the breakthrough curve has reached a plateau.

### 3. Results and discussion

#### 3.1. Effects of flow velocity

The transport of HA in porous media is primarily controlled by the attachment of HA to the solid matrix. The transport behavior of HA in columns varies significantly with solid matrices as observed by Wei et al. (2010). Quartz sand provides the lowest retardation factor ( $R$ ), while river sands contain relatively large retardation factors that increase as the average diameter decreases (Table 2), consistent with findings by Wei et al. (2010). Since the retardation effect is primarily caused by the adsorption and deposition of HA in solid matrix, we used the equilibrium distribution coefficient,  $K$ , and the deposition rate

coefficient,  $K_d$ , to interpret the difference in the retardation effects of different solid matrices. The  $K$  and  $K_d$  values showed the same trend as the retardation factors (Table 2), supporting the finding that the transport of HA is an adsorption–deposition dominated process (Wei et al., 2010), indicating that  $K_d$ ,  $K$  and  $R$  can be used to explain the behaviors of HA in different porous media.

The breakthrough curves resulting from HA transport at different flow velocities are presented in Fig. 1. It is clear that slow flow delayed the transport of HA in porous media. The HA concentrations detected in the three sand column effluents drastically decreased when the flow velocity decreased at any given pore volume. The decreased effluent HA concentration resulting from the decreased flow velocity was more obvious in the river sand column than in the quartz sand column, while for the river sand columns, a smaller average diameter was associated with a greater decrease in the effluent HA concentration in response to decreased flow velocity.

Fig. 2 shows the corresponding  $F_r$ ,  $K_d$ ,  $R$  and  $K$  values as a function of flow velocity for the three sand columns. Although the breakthrough curves had similar changes in patterns with flow velocity, quartz sand and river sand showed quite different

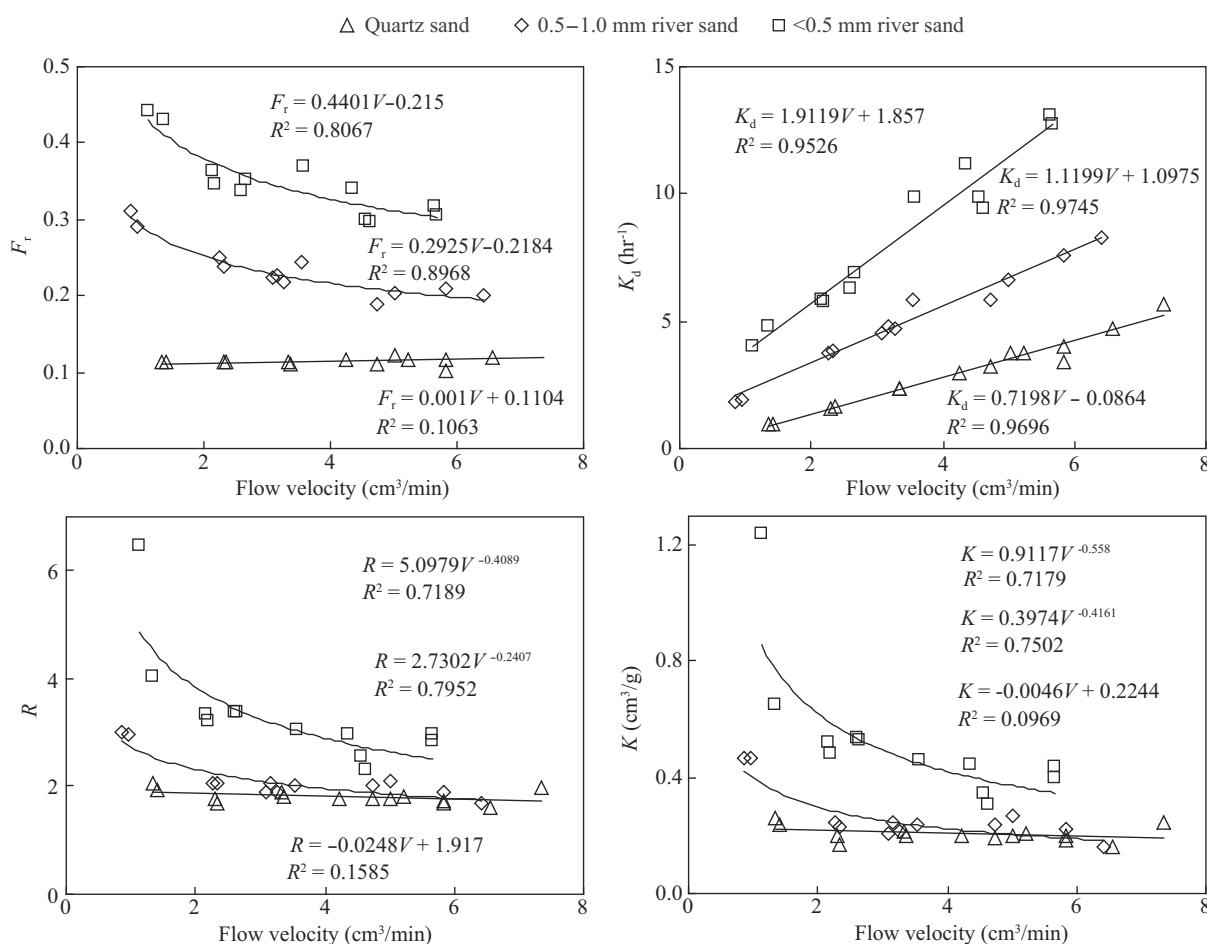


Fig. 2 – Relationship between flow velocity ( $V$ ) and fraction of HA retained in columns ( $F_r$ ), deposition rate coefficient ( $K_d$ ), retardation factor ( $R$ ) and equilibrium distribution coefficient ( $K$ ) for the quartz sand columns, 0.5–1.0 mm river sand columns and <0.5 mm river sand columns. The solution pH was 8 and ionic strength was 0.001 mol/L KCl for all samples.

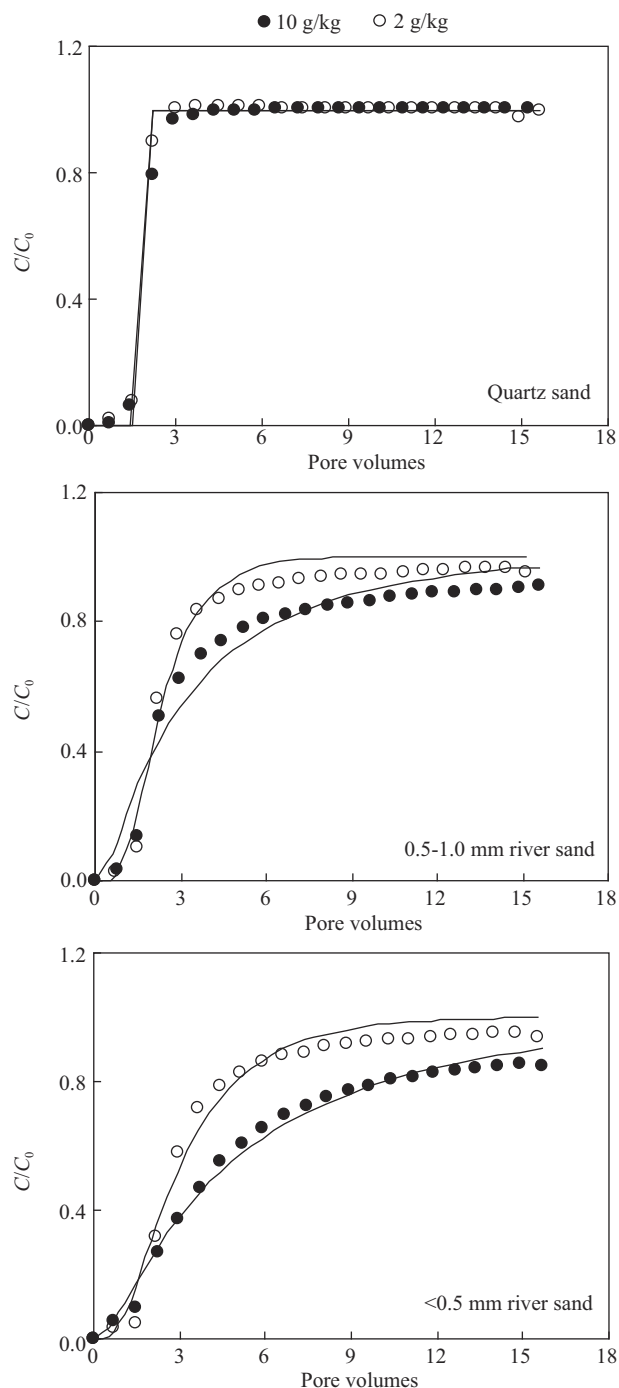
changes in the patterns of  $F_r$ ,  $R$  and  $K$  in response to the flow velocity changes. Specifically, the three parameters all decreased as the flow velocity increased. However, this decrease was exponential in both the 0.5–1 mm and <0.5 mm river sand columns, while it was linear in the quartz sand column. Increasing the flow velocity in porous media decreases the collision time of HA with solid particles (Lecoanet and Woesner, 2004; Kampel et al., 2009), which was likely responsible for the reduced HA absorption that was observed in the columns. Accordingly, the delay in the breakthrough curves was alleviated as illustrated by the changes in  $F_r$ ,  $R$  and  $K$  shown in Fig. 2. For the <0.5 mm river sand column, increasing the flow velocity from 1.12 to 5.68 cm<sup>3</sup>/min resulted in approximately 45%, 128% and 210% decreases in  $F_r$ ,  $R$  and  $K$ , respectively. For the 0.5–1.0 mm river sand column, the decrease in the three parameters was 56%, 79% and 189%, respectively, when the flow velocity increased from 0.86 to 6.43 cm<sup>3</sup>/min. Although  $F_r$  increased slightly as the flow velocity increased from 1.33 to 6.57 cm<sup>3</sup>/min in the quartz sand column, the  $R$  and  $K$  decreased by about 27% and 66%, respectively. Therefore, the high flow velocity induced enhanced HA transport through both the quartz sand and river sand columns were primarily exerted through decreased HA retention in the columns.

Unlike  $F_r$ ,  $R$  and  $K$ , the deposition rate coefficients,  $K_d$ , all increased with flow velocity for both quartz sand and river sand columns. This relationship was also observed by Song and Elimelech (1993) and Kretzschmar et al. (1997) in studies of colloidal particle transport through porous media. In the present study,  $K_d$  increased with flow velocity in a linear relationship rather than a power function as reported by Song and Elimelech (1993). Moreover, the slopes of the corresponding straight lines varied greatly among the solid matrices and followed the order of <0.5 mm river sand > 0.5–1.0 mm river sand > quartz sand, suggesting that  $K_d$  increased more rapidly in the <0.5 mm river sand column than in the quartz sand column when the flow velocity increased.

### 3.2. Effects of influent concentration

The transport behavior of HA at different influent concentrations is shown in Fig. 3. The lower the influent HA concentration, the less HA was transported through the porous media, and accordingly the smaller the  $C/C_0$  in the effluent solution. These findings were consistent conceptually and experimentally with observations by many other authors for the transport of solute (Hanna et al., 2009; Li et al., 2009; Lazaridis and Keenan, 2010), colloids (Zhuang et al., 2005; Frimmel et al., 2007; Hristovski et al., 2008; Liu et al., 2009; Peralta-Videa et al., 2010) and microorganisms (Marlow et al., 1991; Tufenkji et al., 2003; Zhuang and Jin, 2003), suggesting that the effects of influent HA concentrations on HA transport follow the general patterns with the effects on the transport of chemicals, colloids and microorganisms. For the <0.5 mm and 0.5–1.0 mm river sand columns, the  $C/C_0$  values observed when the HA concentration was low were slightly higher than the values observed under high HA concentration conditions for the first 1.5 pore volumes due to the rapid initial attachment of HA molecules to solid particles. However, for subsequent pore volumes, the  $C/C_0$  values were much larger under high HA concentrations than low HA concentrations. This phenomenon may be explained

by the adsorption process during HA transport through the columns since the transport was dominated by adsorption (Wei et al., 2010). At the beginning of the experiment, more HA was injected into the columns; therefore, the collision probability was greater and the amount of HA adsorbed/deposited was



**Fig. 3 – Breakthrough curves of HA in the quartz sand column, 0.5–1.0 mm river sand column and <0.5 mm river sand column at different initial HA concentrations. The solution pH was 8 and ionic strength was 0.001 mol/L for all samples. The flow velocities were 4.73, 2.26 and 3.16 cm<sup>3</sup>/min for the quartz sand column, 0.5–1.0 mm river sand column and <0.5 mm river sand column, respectively.**



greater due to the rapid initial attachment of HA molecules to the solid particles. This resulted in less HA being left in the column solutions. As a result, the  $C/C_0$  was low under high influent HA concentration conditions at the beginning of the transport experiment. However, with the continuous injection of HA, the sorption sites were quickly saturated by the high HA concentration, resulting in the  $C/C_0$  increasing and a plateau in the breakthrough curves being attained. When low concentrations of HA were injected into the columns, less HA was collected and attached to the particles, which resulted in the residual HA concentration in the solution and the  $C/C_0$  in the effluent solution being quite high. Despite this, the adsorption/deposition sites in porous media were saturated slowly, and the adsorption/deposition took a rather long time to reach equilibrium under these conditions, which corresponds to the low  $C/C_0$  in the later transport stage when compared with the value observed under high HA concentration conditions.

The effects of influent HA concentration on HA transport in the columns can also be supported by the parameters shown in Table 3. When compared with the low influent HA, even though more HA collided with the solid matrix and was collected by solid particles when the influent HA was high, the fraction retained by the solid matrix was lower than that retained under low HA concentration conditions due to the large influent HA concentration. As a result, the  $F_r$  was smaller when the HA concentration was high than when it was low.

Although the parameters presented in Table 3 all increased as the influent HA concentration decreased, these increases varied among the solid matrices. The greatest increase in the parameters caused by decreased influent HA concentration was observed in the <0.5 mm river sand columns, in which the  $F_r$ ,  $R$ ,  $K_d$  and  $K$  increased by 58%, 75%, 30% and 15%, respectively. The lowest increase in the parameters was observed in the quartz sand columns, in which the  $F_r$ ,  $R$ ,  $K_d$  and  $K$  increased by 13%, 21%, 2% and 2%, respectively, as the influent HA concentrations decreased from 10 to 2 g/kg. Therefore, the effects of influent HA concentration on HA transport were primarily exerted through changing HA retention in the columns.

The HAs contain both hydrophobic and hydrophilic moieties as well as many functional groups such as carboxylic, phenolic, carbonyl and hydroxyl groups connected to aliphatic or aromatic carbons (Hering and Morel, 1988). The existence of such carboxylic and phenolic groups results in HA being predominantly negatively charged in aqueous solutions (Cornel et al.,

1986). The adsorption of HA to the surfaces of porous media is dependent on selective functional groups of the porous media. Understanding selective adsorption of the functional groups will provide additional information about the mechanisms behind the transport and retention of HA in porous media. Therefore, we recommend in future investigations that functional groups be further examined before and after column experiments by measuring the  $SUVA_{254}$  and/or calculating the  $UV_{254}/TOC$  values.

#### 4. Conclusions

The transport of HA in porous media was affected by the nature of the solid matrix, flow rate and initial HA concentration. Increasing the flow rate and the initial HA concentration accelerated the transport of HA in all of the porous media evaluated. However, the effects of flow rate and initial HA concentration varied among porous matrices and were more notable in sands with smaller average particle diameters. The equilibrium distribution coefficient ( $K$ ), deposition rate coefficient ( $K_d$ ), fraction of HA retained in columns ( $F_r$ ) and retardation factors ( $R$ ) showed similar responses to flow velocity and HA concentration, and could be used to explain the effects of such factors on HA attachment in solid matrices and the transport of HA through porous media.

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**Table 3 – Effects of influent HA concentration on various parameters.**

|                       | Influent concentration<br>(g/kg) | Flow velocity<br>(cm <sup>3</sup> /min) | $F_r$ | $K_d$<br>(hr <sup>-1</sup> ) | $R$  | $K$<br>(cm <sup>3</sup> /g) |
|-----------------------|----------------------------------|---|-------|------------------------------|------|-----------------------------|
| Quartz sand           | 10                               | 4.73                                    | 0.104 | 3.00                         | 1.80 | 0.197                       |
| Quartz sand           | 2                                | 4.73                                    | 0.117 | 3.64                         | 1.83 | 0.201                       |
| 0.5–1 river sand      | 10                               | 2.26                                    | 0.167 | 2.45                         | 1.86 | 0.201                       |
| 0.5–1 river sand      | 2                                | 2.26                                    | 0.255 | 3.98                         | 1.90 | 0.210                       |
| <0.5 mm river sand    | 10                               | 3.16                                    | 0.228 | 4.83                         | 2.28 | 0.287                       |
| <0.5 mm<br>river sand | 2                                | 3.16                                    | 0.360 | 8.45                         | 2.98 | 0.330                       |

$F_r$ : the fraction of HA retained in the column;  $K_d$ : the deposition rate coefficient;  $R$ : the retardation factor;  $K$ : the equilibrium distribution coefficient.

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