Designing laboratory metallic iron columns for better result comparability

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Abstract

Despite the amount of data available on investigating the process of aqueous contaminant removal by metallic iron (Fe\textsuperscript{0}), there is still a significant amount of uncertainty surrounding the design of Fe\textsuperscript{0} beds for laboratory testing to determine the suitability of Fe\textsuperscript{0} materials for field applications. Available data were obtained under various operating conditions (e.g. column characteristics, Fe\textsuperscript{0} characteristics, contaminant characteristics, oxygen availability, solution pH) and are hardly comparable to each other. The volumetric expansive nature of iron corrosion has been univocally reported as major drawback for Fe\textsuperscript{0} beds. Mixing Fe\textsuperscript{0} with inert materials has been discussed as an efficient tool to improve sustainability of Fe\textsuperscript{0} beds. This paper discusses some problems associated with the design of Fe\textsuperscript{0} beds and proposes a general approach for the characterization of Fe\textsuperscript{0} beds. Each Fe\textsuperscript{0} column should be characterized by its initial porosity, the composition of the steady phase and the volumetric proportion of individual materials. Used materials should be characterized by their density, porosity, and particle size. This work has introduced simple and reliable mathematical equations for column design, which include the normalisation of raw experimental data prior to any data treatment.

Key words: Deep bed filtration, Operational parameters, Results comparability, Water Filtration; Zerovalent iron.
Metallic iron (Fe\textsuperscript{0}) as reactive medium for aqueous contaminant removal has been intensively investigated during the last two decades [1-6]. These studies have demonstrated the potential of Fe\textsuperscript{0} for use in (i) subsurface permeable reactive barriers [1], (ii) above ground wastewater treatment [3,5], (iii) household water filters [7-10], and (iv) drinking water treatment plants [11-13]. The fundamental mechanisms of contaminant removal in Fe\textsuperscript{0} beds are adsorptive size-exclusion and co-precipitation [5,6,13-19].

Data for pilot- and full-scale remediation scheme are mostly obtained from laboratory columns [20-22]. Laboratory column studies are in turn conventionally designed based on batch treatability studies [21,23,24]. A major problem of available data from batch studies is the poor comparability of results from different laboratories using different conditions. Most experimental conditions are not relevant for field situations [25,26]. For example, only shaking intensity lower than 50 m\textsuperscript{-1} could enable the formation of a universal oxide-scale in the vicinity of the Fe\textsuperscript{0} surface as observed in column studies and in full-scale barriers [25]. A careful look behind published data on laboratory column experiments ([27-32]; see Tab. 1) also demonstrates large variability in the experimental design. General design procedures are not available. Differences in design procedures may cloud interpretations of reported data. Therefore, any effort to ensure the comparability of laboratory results over time and space would accelerate the development of the Fe\textsuperscript{0} remediation technology.

The objective of the present communication is to improve the comparability of laboratory column results by offering the scientific community tools for a common basis for the design of Fe\textsuperscript{0} beds. Previous theoretical works [9,31,32] have demonstrated that mixing Fe\textsuperscript{0} and other materials (gravel, pumice, sand, quartz) is the prerequisite for long service life. This mixture should be characterized by the volumetric proportion of Fe\textsuperscript{0} (Fig. 1) and other materials (additives) and the initial porosity [32]. The specific objective of this communication is to establish equations for the evaluation of the mass of the materials to be used.
2 Basis for comparable results

Cylindrical columns usually used for laboratory experiments primarily differ in their size (diameter * length). For example, columns with 5 cm internal diameters exist with lengths varying from 30 to 100 cm (Tab. 1). Completely filling the six different columns from Tab. 1 with reactive materials (e.g. Fe\textsuperscript{0}) will yield 0.2 to 9.8 kg of Fe\textsuperscript{0} for the experiment. The question is how to objectively compare results obtained with various amount of the same materials? The situation is exacerbated when the materials are different (particle size, shape, chemical composition or generally intrinsic reactivity). When material mixture should be performed, the rationale for purposeful mixing should be developed.

Using the material apparent specific weight tabulated by Noubactep and Caré [9], Tab. 1 clearly shows that a given volume (e.g. V\textsubscript{solid} in the same column) is occupied by a mass m of pumice, 4.14*m of sand, and 12.19*m of Fe\textsuperscript{0}. The coefficients are the ratios of the apparent specific weights (\(\rho_i/\rho_j\)). This observation confirms that the volumetric ratio is the sole appropriate approach to design column studies. Accordingly, an approach to have comparable results could be to fix the Fe\textsuperscript{0} volume. A given Fe\textsuperscript{0} volume corresponds to various heights depending on the column internal diameter. Accordingly, beside used Fe\textsuperscript{0} mass (corresponding to a volumetric fraction of solid), the column dimensions should always be specified. Three examples are given below for illustration.

In the laboratory, the Fe\textsuperscript{0} amount is commonly given in weight (g or kg). Therefore, a practical approach is to fix a mass of Fe\textsuperscript{0} \((m_0)\) which is likely to enable observable effects within a reasonable time (e.g. 6 weeks).

- An experiment is designed to compare the reactivity of different Fe\textsuperscript{0} materials. Parallel column experiments have to be conducted in which Fe\textsuperscript{0} is the sole variable and the same mass \((m_0)\) is used in individual columns.

- An experiment is designed to compare the impacts of chloride (Cl\textsuperscript{-}) and sulphates (SO\textsubscript{4}\textsuperscript{2-}) on the efficient of a Fe\textsuperscript{0} for methylene blue (MB) discoloration. The three following parallel
experiments could be conducted with the mass \( m_0 \) of a Fe\(^0 \) sample: (i) MB in deionised water, (ii) MB in a Cl\(^- \) solution, and (iii) MB in a SO\(_4^{2-} \) solution. The MB concentration is the same in all experiments, Cl\(^- \) and SO\(_4^{2-} \) solutions have the same equivalent concentration.

- An experiment is designed to compare the impact of mixing Fe\(^0 \) with additives. The reference system should be the mass \( m_0 \) of Fe\(^0 \), and volumetric proportions of Fe\(^0 \) have to be replaced by appropriated additives. Tab. 1 shows that each column is characterized by a constant pore volume which is the external or inter-granular porosity. For porous materials the internal porosity or intra-granular porosity has been discussed as a storage room for in-situ generated corrosion products and thus a tool to sustain Fe\(^0 \) reactivity [9,10,32]. Similarly, mixing Fe\(^0 \) and non-porous inert additives is beneficial for the system because inert material will not contribute to pore filling or filter clogging.

The next section will establish some general equations to support design operations.

### 3 Mathematical equations for column design

A hypothetical cylindrical filter having an inner diameter \( D \), a length \( L \), and a reactive zone \( h_{rz} \) is considered. The reactive zone is the fraction of \( L \) (\( L > h_{rz} \)) containing the reactive material, possibly mixed with selected additives. The filter is filled by spherical particles (reactive materials and non-reactive additives) having a constant diameter \( d \). Considering the granular material as composed of mono-dispersed spheres subjected to soft vibrations, the column compactness (or packing density) \( C \) ranges between 0.60 and 0.64 for a random close packing but it is generally considered to be equal to 0.64 (limit value). It can be noticed that the value of the compactness depends on various parameters as the distribution size of particles, their shape [33-34]. The theoretical value of \( C = 0.64 \) is strictly valid for particles with spherical shape and similar sizes. It is assumed in this study that ratio of cylinder diameter (\( D = 2*R \)) to particle diameter (\( d \)), \( \beta = 2*R/d \) is optimal for axial hydrodynamic dispersion [36,37].
The volume of the reactive zone ($V_{rz}$), the volume of solid ($V_{solid}$), the volume of inter-
granular pores ($V_{pore}$), the volume of individual solids ($V_i$) with the apparent specific weight
$\rho_i$, and the thickness of the reactive zone ($h_{rz}$) are given by Eq. 1 through Eq. 6:

1. $V_{rz} = \pi D^2 h_{rz}/4$  \hspace{1cm} (1)
2. $V_{rz} = V_{solid} + V_{pore}$  \hspace{1cm} (2)
3a. $V_{solid} = C V_{rz}$  \hspace{1cm} (3a)
3b. $V_{pore} = (1 - C) V_{rz}$  \hspace{1cm} (3b)
4. $V_{solid} = \sum V_i = V_{solid1} + V_{solid2} + V_{solid3} + \ldots$  \hspace{1cm} (4)
5. $V_i = m_i/\rho_i$  \hspace{1cm} (5)
6. $h_{rz} = 4V_{rz}/\pi D^2 = 4V_{solid}/(C \pi D^2)$  \hspace{1cm} (6)

### 3.1 Volumetric solid fractions in the reactive zone

Replacing $V_{solid}$ in Eq. 2 by its expression from Eq. 4 yields:

$$V_{rz} = V_{solid} + V_{pore} + \sum V_i \Rightarrow \alpha_{pore} + \sum \alpha_i = 1$$  \hspace{1cm} (7)

where $\alpha_{pore}$ and $\alpha_i$ are the volumetric fractions of the inter-granular pores and of the individual
solid phase “i” in the column respectively. The fractions are related to the volume of the
reactive zone $V_{rz}$: ($\alpha_{pore} = V_{pore}/V_{rz}$ and $\alpha_i = V_{solid_i}/V_{rz}$).

Eq. 7 suggests that $\sum \alpha_i$ is necessarily equal to C. Thus, Eq. 2 should be written:

$$V_{rz} = V_{solid} + V_{pore} = V_{rz} (\alpha_{pore} + \sum \alpha_i)$$  \hspace{1cm} (8)

For instance, for a system containing a mixture of four solid phases, Eq. 7 should read:

$$\alpha_{pore} + \alpha_1 + \alpha_2 + \alpha_3 + \alpha_4 = 1$$  \hspace{1cm} (8a)
$$\alpha_1 + \alpha_2 + \alpha_3 + \alpha_4 = 1 - \alpha_{pore} = C$$  \hspace{1cm} (8b)

Eq. 8 is very important for the calculations of the amounts of individual additives to achieve
wished material mixtures as will be discussed in the next section. Eq. 8b suggests that $\alpha_i$ is
necessarily a fraction of C. Thus, a simple rule of proportion can be established to calculate $\alpha_i$.
for each proportion $P_i$ of the solid phase. $C$ necessarily corresponds to 100 % solid. The
relation between $\alpha_i$ and $P_i$ is given by Eq. 9:

$$\alpha_i = C*P_i / 100 \quad (9)$$

Tab. 2 gives some values of $\alpha_i$ for selected relevant $P_i$ values. For example, if a quaternary
system should be made up of 40 % of material A, 30 % material B, 20 % material C and 10 %
material D, the coefficients to be used are: 0.256, 0.192, 0.128 and 0.064 respectively. A
quaternary system seems to be strange or complicated. However, such a system could
comprise $\mathrm{Fe}^0$ as basic reactive material, $\mathrm{MnO}_2$ to sustain $\mathrm{Fe}^0$ reactivity, pumice as storage
solid and quartz as non-porous inert filling material.

3.2 Initial porosity of reactive the zone

In the case of non porous spherical particles, the initial porosity of the reactive zone $\Phi_0$ is the
inter-granular porosity $\alpha_{\text{pore}}$ and is given by Eq. 10 [32].

$$\Phi_0 = 1 - C \quad (10)$$

If individual particles are porous, the intra-granular porosity should be considered [32]. In this
case the initial porosity should read:

$$\Phi_0 = (1 - C) + \Sigma \alpha_i \phi_i \quad (11)$$

where $C$ is the compactness of the reactive zone, $\alpha_i$ the volumetric fraction of the solid i (Tab.
2) and $\phi_i$ its internal porosity.

Equations 1 to 11 are the basis for purposeful experimental design as will be discussed below
for selected cases.

4 Designing some $\mathrm{Fe}^0$ systems

4.1 A four component system

For the design of a quaternary system of $\mathrm{Fe}^0$, sand, pumice and $\mathrm{MnO}_2$, Eq. 8 can be used:

$$V_{rz} = V_{rz} (\alpha_{\text{pore}} + \alpha_{\text{Fe}} + \alpha_{\text{sand}} + \alpha_{\text{pumice}} + \alpha_{\text{MnO}_2})$$

$$V_{rz} = \alpha_{\text{pore}} \cdot V_{rz} + \alpha_{\text{Fe}} \cdot V_{rz} + \alpha_{\text{sand}} \cdot V_{rz} + \alpha_{\text{pumice}} \cdot V_{rz} + \alpha_{\text{MnO}_2} \cdot V_{rz}$$
\[ V_{rz} = V_{pore} + V_{Fe} + V_{sand} + V_{pumice} + V_{MnO2} \]

Assuming \( V_{rz} = 1000 \text{mL} \), \( \alpha_{Fe} = 0.192 \) (\( P_{Fe} = 30\% \)), \( \alpha_{sand} = 0.256 \) (\( P_{sand} = 40\% \)), \( \alpha_{pumice} = 0.128 \) (\( P_{pumice} = 20\% \)), and \( \alpha_{MnO2} = 0.064 \) (\( P_{MnO2} = 10\% \)), the volume of the materials are 192, 256, 128 and 64 mL respectively and \( V_{pore} = 360 \text{mL} \).

Using Eq. 5 \( (m_i = \rho_i * V_i) \), the needed mass of each material can be calculated. Results showed that 1498 g of Fe, 678 g of sand, 82 g of pumice and 224 g of MnO\(_2\) will be used and homogeneously mixed to design the desired column.

The initial porosity of the resulted column is given by Eq. 11

\[ \Phi_0 = (1 – C) + \alpha_{Fe} \phi_{Fe} + \alpha_{sand} \phi_{sand} + \alpha_{pumice} \phi_{pumice} + \alpha_{MnO2} \phi_{MnO2} \]

But Fe and sand (quartz) are non-porous, therefore \( \Phi_0 \) is given by:

\[ \Phi_0 = (1 – C) + \alpha_{pumice} \phi_{pumice} + \alpha_{MnO2} \phi_{MnO2} \]

Using values above \( (\alpha_{pumice} = 0.128 \text{ and } \alpha_{MnO2} = 0.064) \), \( \phi = 0.80 \) (80\%) for pumice, and \( \phi = 0.25 \) (25\%) for MnO\(_2\), give an initial porosity of 0.48 (vs. 0.36 in a pure Fe\(^0\) column).

This example shows an increase of the initial porosity by 33\% for the given mixture.

### 4.2 Working with a constant Fe\(^0\) mass

To work with a constant Fe\(^0\) mass, the volume \( (V_{Fe}) \) occupied by the given Fe mass is calculated using Eq. 5. This volume represents a certain fraction of solid in the filter \( (\alpha_{Fe} \text{ in Eq. 8a}) \). Knowing \( V_{solid} \), the volume of the reactive zone \( V_{rz} \) and its thickness \( h_{rz} \) can be deduced using Eq. 3c and Eq. 6.

It is obvious, that \( h_{rz} \) depends on the inner diameter of the filter. Accordingly, the simplest way to investigate the effect of bed thickness on the efficiency of Fe\(^0\) beds is to work with a constant Fe\(^0\) mass, representing a fix volumetric percentage of solid and using columns of various internal diameters \( (D_i) \).

Working with a constant Fe\(^0\) mass can be regarded as the most powerful tool to achieve comparable results. For example, laboratory column experiments could be routinely
performed with 100 g of Fe\(^0\) representing 30 vol-% of the solid in the reactive zone. Calculations show that the reactive zone will occupy a volume of 20 mL. To have such a volume using columns with inner diameters D\(_1\) (2.0 cm), D\(_2\) (2.6 cm) and D\(_3\) (5.0 cm), the column length should be 6.37, 3.77, and 1.02 cm respectively. All columns of Tab. 1 satisfy this basic condition. However building an homogeneous 1.02 cm layer of “Fe\(^0\) + additive” could be a difficult task. While repeating the calculations above with 250 g Fe\(^0\) results show that needed column lengths are 15.93, 9.43, and 2.55 cm respectively. While column 1 (D = 2 cm, L = 12 cm) is no more applicable, filling column 3 (D = 5 cm) with only 2.55 cm of homogeneous mixture could still be difficult to achieved. Therefore an objective could be a minimum reactive zone length of 5 cm. To achieve this thickness, a Fe\(^0\) of 500 g is needed for column 3. This is necessarily coupled with longer experimental duration. As a result, it is suggested that columns with more than 3 cm internal diameter are not suitable for short term laboratory experiments. This suggestion is coupled with the desire to save Fe\(^0\) costs in columns and to shorten experimental durations.

### 4.3 Working with a constant initial porosity

For column experiments performed with a constant Fe\(^0\) mass (e.g. 100 g), using various additives could enable a constant initial porosity (\(\Phi_0\)). The aim could be for example to experimentally quantify the impact of MnO\(_2\) on Fe\(^0\) bed’s hydraulic permeability. The length of the reactive zone will vary as a function of the porosity of the tested additive. Eq. 11 (\(\Phi_0 = (1 – C) + \Sigma \alpha_i \phi_i\)) should be solved while fixing one or two variables.

For example, the reference system uses Fe\(^0\) (100 g representing 50 vol-%) and a limestone with \(\phi_{lime} = 25\%\). A second system having the same initial porosity (\(\Phi_0\)) and been made up of Fe (\(\phi_{Fe} = 0\%\)), quartz (\(\phi_{quartz} = 0\%\)) and pumice should be designed. Which pumice amount should be used?
For the reference system, $\alpha_{\text{Fe}} = 0.32; \phi_{\text{Fe}} = 0; \alpha_{\text{lime}} = 0.32; \text{and } \phi_{\text{lime}} = 0.25$. Calculations give $\Phi_0 = 1 - C + 0.32 \times 0.25 = 0.44$. Now, Eq. 11 ($\Phi_0 = 0.44$) should be written for the unknown system and solved for $\phi_{\text{pumice}}$, given $\alpha_{\text{Fe}} = 0.32 \text{ (or } \alpha_{\text{quartz}} + \alpha_{\text{pumice}} = 0.32)$. That is, with $\phi_{\text{Fe}} = \phi_{\text{quartz}} = 0$:

$$\Phi_0 = 0.44 = 1 - C + \alpha_{\text{Fe}} \phi_{\text{Fe}} + \alpha_{\text{quartz}} \phi_{\text{quartz}} + \alpha_{\text{pumice}} \phi_{\text{pumice}} \quad (11a)$$

$$\alpha_{\text{pumice}} = 0.08/\phi_{\text{pumice}} \quad (11b)$$

The pumices to be used are materials for which Eq. 11b and the fundamental conditions $\alpha_{\text{quartz}} + \alpha_{\text{pumice}} = 0.32$ and $\phi_{\text{pumice}} < 0.9$ are respected. There are certainly a large number of possible solutions but the appropriate solution will be dictated by pumice’s availability. Table 3 summarizes 5 appropriate solutions corresponding to $\phi_{\text{pumice}}$ values varying from 0.33 to 0.80.

Having the $\alpha_{\text{pumice}}$ (and $\alpha_{\text{quartz}}$), the volumes and the masses can be derived and used as described above.

5 Concluding remarks

To date, the equivocal results published on contaminant removal in $\text{Fe}^0/\text{H}_2\text{O}$ systems demonstrate that the physico-chemical mechanisms of the remediation process still remain unclear [38]. Available data were produced and interpreted based on the concept that $\text{Fe}^0$ is a reducing agent [1-4]. On the other side, available data are characterized by the diversity of experimental conditions under which they are obtained (Tab. 1). This situation is not favourable for reliable data comparison. In particular, further laboratory column studies aiming at optimising field $\text{Fe}^0$ bed design efforts should be acquired under reproducible experimental conditions.

The present theoretical study has initiated a new path to an harmonized experimental protocol for column experiments. It is proposed that 100 g or 250 g of $\text{Fe}^0$ is used in a volumetric proportion not larger than 50 %. The equations for material mixtures are established. It is hoped that researchers will use the developed tools to avoid inconsistent results which are
most likely the product of inaccurate experimental designs. For example, while bed clogging has been reported as the major drawback of the Fe\textsuperscript{0} technology, most researchers have used experimental designs favouring column clogging (Fe\textsuperscript{0} > 60 vol-%) [32].

Once the mass of Fe\textsuperscript{0} and its volumetric proportion are fixed several other factors can be purposefully discussed. For example, the impact of: (i) the Fe\textsuperscript{0} average particle size, (ii) the solution pH, (iii) the water composition, (iv) water flow velocity. In all the case the description of the experimental protocol should include and extensive description of used materials (density, porosity, measured bed porosity). Finally, because clogging is the major problem of Fe\textsuperscript{0} beds, expertise from other branches of environmental science dealing with porous media clogging could be helpful for proper Fe\textsuperscript{0} bed design. In particular, much impetus could come from the impact of bio-clogging on the evolution of the porosity of constructed wetland [39-42].

References


**Table 1:** Variability of the operational conditions for column experiments as illustrated by material masses likely to be used in six different columns. D is the internal diameter of the column and L its length. It is assumed that the entire column volume is filled by spherical particle of Fe, sand or pumice in an ideal cubic packing (C = 0.64).

<table>
<thead>
<tr>
<th>D (cm)</th>
<th>L (cm)</th>
<th>V&lt;sub&gt;column&lt;/sub&gt; (cm³)</th>
<th>V&lt;sub&gt;solid&lt;/sub&gt; (cm³)</th>
<th>m&lt;sub&gt;Fe&lt;/sub&gt; (kg)</th>
<th>m&lt;sub&gt;sand&lt;/sub&gt; (kg)</th>
<th>m&lt;sub&gt;pumice&lt;/sub&gt; (kg)</th>
<th>Ref.</th>
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<tr>
<td>2.0</td>
<td>12</td>
<td>37.7</td>
<td>24.1</td>
<td>0.19</td>
<td>0.06</td>
<td>0.015</td>
<td>[27]</td>
</tr>
<tr>
<td>2.6</td>
<td>40</td>
<td>212.5</td>
<td>136.0</td>
<td>1.06</td>
<td>0.34</td>
<td>0.087</td>
<td>[28]</td>
</tr>
<tr>
<td>4.1</td>
<td>25</td>
<td>330.0</td>
<td>211.0</td>
<td>1.60</td>
<td>0.60</td>
<td>0.135</td>
<td>[29]</td>
</tr>
<tr>
<td>5.0</td>
<td>30</td>
<td>589.3</td>
<td>377.1</td>
<td>2.94</td>
<td>0.94</td>
<td>0.241</td>
<td>[30]</td>
</tr>
<tr>
<td>5.0</td>
<td>50</td>
<td>982.1</td>
<td>628.6</td>
<td>4.90</td>
<td>1.57</td>
<td>0.402</td>
<td>[31]</td>
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<tr>
<td>5.0</td>
<td>100</td>
<td>1964.3</td>
<td>1257.1</td>
<td>9.81</td>
<td>3.14</td>
<td>0.805</td>
<td>[32]</td>
</tr>
</tbody>
</table>
Table 2: Relation between the volumetric fraction ($\alpha_i$) of solid materials in the column ($\alpha_{pore} = 0.36$) and their volumetric percent ($P_i$) as solid. $\alpha_i$ is obtained by a rule of proportion relative to ($\alpha_{max} = 0.64$ corresponding to 100 % solid). $\alpha$ values are very useful for multi-solid system designs (see text).

<table>
<thead>
<tr>
<th>$P_i$</th>
<th>0</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>40</th>
<th>50</th>
<th>60</th>
<th>70</th>
<th>80</th>
<th>90</th>
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<tr>
<td>$\alpha_i$</td>
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<td>0.064</td>
<td>0.128</td>
<td>0.192</td>
<td>0.256</td>
<td>0.320</td>
<td>0.384</td>
<td>0.448</td>
<td>0.512</td>
<td>0.576</td>
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Table 3: selected appropriate solutions for Eq. 11b.

<table>
<thead>
<tr>
<th></th>
<th>( \alpha_{\text{quartz}} )</th>
<th>( \alpha_{\text{pumice}} )</th>
<th>( \phi_{\text{pumice}} )</th>
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<tbody>
<tr>
<td></td>
<td>0.08</td>
<td>0.24</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>0.13</td>
<td>0.19</td>
<td>0.42</td>
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<tr>
<td></td>
<td>0.16</td>
<td>0.16</td>
<td>0.50</td>
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<td></td>
<td>0.19</td>
<td>0.13</td>
<td>0.62</td>
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<tr>
<td></td>
<td>0.22</td>
<td>0.1</td>
<td>0.80</td>
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Figure 1: Evolution of the material weight percent in a ternary system Fe\textsuperscript{0}/sand/pumice as the initial 50:50 sand:pumice mixture (vol) is progressively amended with Fe\textsuperscript{0}. The sand:pumice volumetric ratio is kept constant during the whole simulation. It is evident that due to difference in densities the weight percent variation is different for individual materials.