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EXPERIMENTAL CHARACTERIZATION OF CERAMIC SPONGES AS COLUMN INTERNALS

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Abstract
Ceramic sponges are monolithic network structures with high specific surface areas at a low pressure drop compared to other ceramic column internals. The characterization of hydrodynamics as well as mass transfer in two phase counter-current flow is the basis for the comparison of sponges with other packings. Sponges of different material, pore size and porosity are thus evaluated concerning holdup, pressure drop and flooding behavior as well as gas and liquid side mass transfer coefficients and interfacial surface areas.

Keywords: ceramic sponges, column packings, hydrodynamics, mass transfer, effective interfacial area

1. Introduction

Ceramic sponges are monolithic network structures with two continious phases: solid and void phase. They are often still called "open-celled foams" in literature, although foams consist of closed cells. "Sponge" seems more appropriate for this new class of materials in chemical engineering and is thus used in this work. Ceramic sponges can be produced in a wide range of materials and geometrical properties, such as pore size, pororsity and sample size, and are commercially available as molten metal filters for foundry technology. They are often produced as replicates of ploymer sponges after the methode of Schwarzwalder[1]. Metallic sponges are also available on the market, but were not investigated in this work.

Monolithic network structures in general are of great interest for various applications in chemical engineering. Ceramic sponges especially qualify by their low pressure drop at high specific surface areas. The values for the specific surface areas lie in the range of those for structured column packings. Sponges are available in a broad variety of ceramic materials, which are often chemically inert for the desired application. Enhanced heat transfer due to the continuous solid phase makes them interesting for heat sensitive applications. Over the last two decades a broad range of possible applications for sponge structures is reported in literature. They cover solar receivers or porous gas burners as well as catalyst supports for various applications such as partial or preferential oxidation, gas purification or Fischer-Tropsch-Sythesis. Reviews on the possible application fields have been provided by different authors[2,3]. Nevertheless, only few investigations on sponges as column internals can be found. Stemmet[4-7] investigated hydrodynamics and mass transfer in counter-current as well as co-current flow through metallic sponge samples. Calvo[8] investigated the liquid holdup in metal sponges and Leveque[9] used ceramic sponges in destillation columns. These authors mostly restrict their work to only a very small range of sample parameters, so the available data for sponges as column internals is comparatively small with a focus on metallic sponge samples.

The aim of this work is thus to characterize and compare ceramic sponge samples of different manufacturers, materials and geometric parameters as column internals experimentally. This included the geometric characterization of the samples as well as the determination of hydrodynamics and mass transfer in counter current flow. Therefore different experimental setups were used to determine the static and dynamic liquid holdup, flooding behavior, gas and liquid side mass transfer coefficients as well as the geometric and the effective specific surface area of sponge samples.

2. Experimental Approach

The results presented in this work were obtained with selected sponge samples. They were purchased from "Vesuvius Becker & Piscantor Grossalmeroder Schmelztiegelwerke GmbH", Germany, in case of the samples made of 99% pure aluminum oxide, and from "Erbicol SA", Switzerland, in case of the silicon infiltrated silicon carbide samples. The nominal properties provided by the manufacturers were porosity and pore number (pores per linear inch, ppi). The pore number increases with decreasing pore size. These data were not detailed enough to ensure comparability of the sponge samples. Therefore further geometrical characterization of the sponges was performed. The samples used were summarized in Table 1. All samples used had a diameter of 100 mm but different sample heights.

Table 1. Samples used

Manufacturer	Porosity	ppi	Sample height in mm	Material	Specific surface area in m²/m³	Amount of closed faces
Vesuvius	0.85	10	50	Al_2O_3	630	Many
Vesuvius	0.75	10	25	Al_2O_3	640	Some
Erbicol	0.88	10	25	SiSiC	480	Little
Erbicol	0.88	20	25	SiSiC	680	Little

2.1 Geometrical characterization

The geometrical characterization of the sponge samples included the determination of light microscopic data, the specific surface area and the porosity. A detailed description of the procedures used can be found in a previous publication[10]. Here only a short summary will be given. Microscopic characterization of sponge samples is well established. The determination of face and strut diameter was performed for all samples. The determination of the porosity was performed with mercury porosimetry. In addition to that the specific surface area was determined with Magnetic Resonance Imaging (MRI). The volume image generated with MRI was processed and evaluated with well established relations from statistical geometry[11].

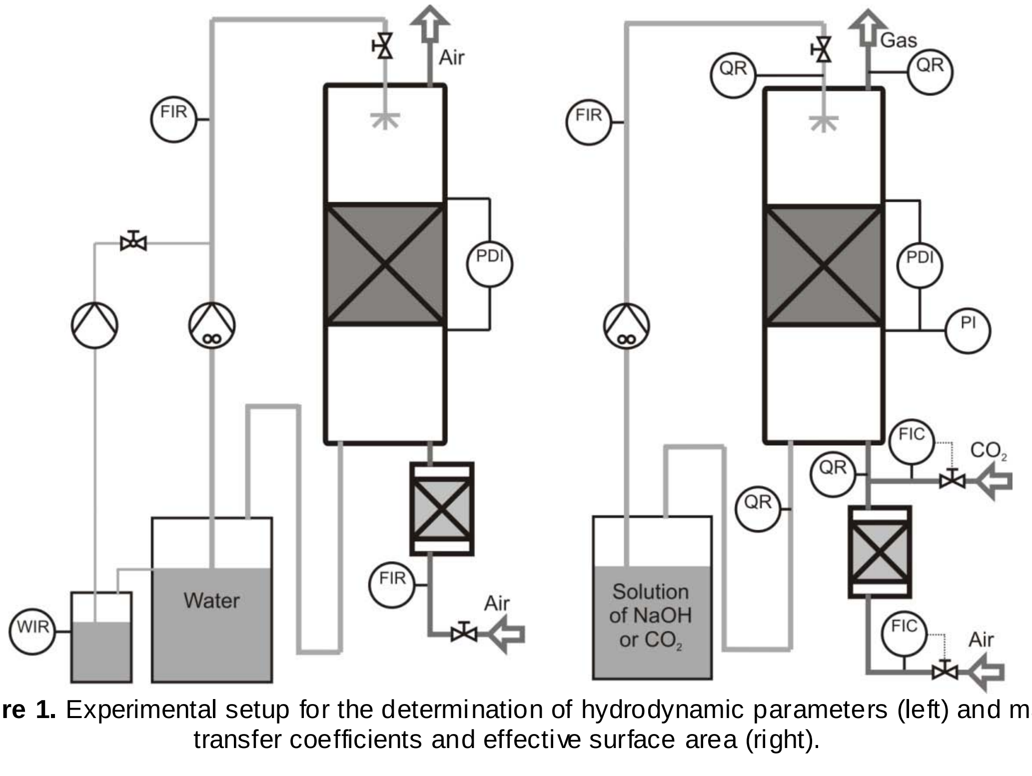

Figure 1. Experimental setup for the determination of hydrodynamic parameters (left) and mass transfer coefficients and effective surface area (right).

2.2 Hydrodynamics

The determination of the hydrodynamic parameters was the main focus of this work. It included dry and wet pressure drop as well as static and dynamic holdup. The holdup measurements also allowed the evaluation of loading and flooding behavior. The test system chosen was water – air, as a broad data basis for this system can be found in literature. Thus a thorough characterization of the hydrodynamics of ceramic sponges in two phase counter-current flow should be possible.

The static liquid holdup was determined using a gravimetric drainage method. The dried sponges were weighed, placed in Plexiglas devices and dipped in water for 5 minutes to ensure a thorough wetting. They were allowed to drain for 15 min in a water-saturated atmosphere and weighed afterwards. From the mass difference of dry and wet sponge the static liquid holdup can be calculated.

The dynamic liquid holdup was determined with the experimental setup shown on the left in Figure 1. Water was circulated through the column with a main pump and distributed by a pipe distributor with 1000-1500 drip points per square meter with 1 mm diameter. All vessels and the hosing were kept full of water during the experiment. The spill of the main vessel led the water in the small tank on the balance. The amount of water in the column could be determined online by pumping a small amount of the liquid load from this tank. The main vessel acted as a buffer for variations in the liquid drainage of the packing. The water-saturated gas was led through a gas distributor pipe with a downward opening of 50 x 13 mm in the column in counter-current flow to the liquid. A differential pressure gauge enabled the measurement of the pressure drop over the packing. Reference experiments without sponge packing in the column allowed to determine the volume in the circuit. The difference of the amount of water in the experiments with and without sponge is equal to the amount of water in the sponge. For all experiments, a packing height of 20 cm was used.

2.3 Mass transfer

For the measurements of the mass transfer coefficients and the specific surface area, the experimental setup was adapted. The method chosen for the determination of the gas-side mass transfer coefficient and the specific surface area was the Dackwerts-plot based on the chemical absorption of carbon dioxide from air in a caustic soda solution with variable concentration, which was established by Wales[12] basing on the work of Danckwerts[13]. The physical stripping of carbon dioxide from water into air was chosen for the liquid-side mass transfer[14].

The experimental setup was chosen similar to the determination of hydrodynamics and is shown in Figure 1 on the right side. The main vessel was filled either with caustic soda solution or water saturated with carbon dioxide. On the gas side the pressurized air saturated with water could be mixed with carbon dioxide for the gas-side mass transfer experiments. Gas-side concentrations were measured with a BRUKER Tensor 27 FT-IR spectrometer. Liquid-side concentrations were measured by acidimetric titration. The packing height chosen for these experiments was 35 cm.

3. Results and Discussion

The results presented in this work were obtained with the selected sponge samples given in Table 1. They were chosen due to their ability to enable counter-current flow of gas and liquid without immediate flooding. The focus was set on the determination of the hydrodynamic parameters. The mass transfer parameters are topic of current research, therefore only first results are presented here.

3.1 Geometrical characterization

The results of the geometrical characterization were already discussed in detail in a previous publication[10]. In Table 1 only the determined specific surface area and the amount of closed faces are given as essential characteristics. The range of the specific surface area of sponge samples is comparable to the values of structured packings. It gives the possibility to compare sponges to other column internals and provides a basis for all further data evaluation with well established correlations for packings. The amount of closed faces stands for the quality of the samples. Here great differences depending on the manufacturing process and the sample size can be observed.

3.2 Hydrodynamics

The measured static holdup values for the selected sponge samples are presented in Figure 2. The error bars indicate the standard deviation of the measured values. Each experiment was performed three times at least. The sponges made of silicon infiltrated silicon carbide have less closed faces and less cavities inside the solid network compared with the alumina samples due to their silicon infiltration. They therefore also have a comparatively low static liquid holdup. In general the holdup of sponges is higher than for structured packings or packed beds, although the values for ceramic materials always scatter[15]. The static liquid holdup increases with decreasing porosity and increasing ppi-number, which is equivalent to a decrease of the pore size. It is also clearly visible, that samples with nearly the same nominal porosity and ppi-number differ in specific surface area as well as static liquid holdup. A direct correlation of specific surface area and holdup is neither possible. Further data such as porosity and sample quality are also of great importance.

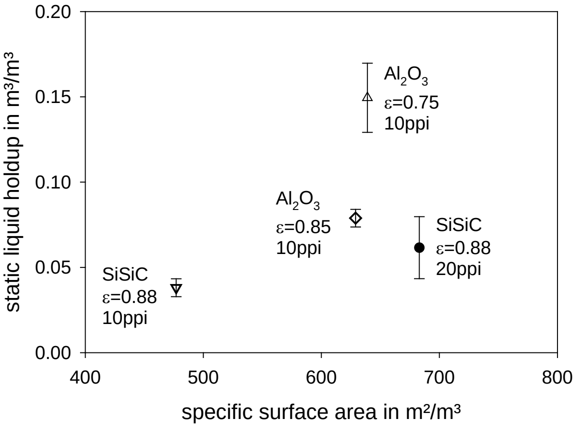

Figure 2. Static liquid holdup of all samples as a function of the specific surface area.

The total liquid holdup and wet pressure drop for one sponge type as a function of the liquid as well as the gas load are presented in Figure 3. As could be expected, the liquid holdup increases with increasing liquid load. Flooding was always observed after the last point indicated in the diagram, which means that the liquid was no longer able to flow out of the sponge. For the selected sponge sample, a loading region could not be observed. The wet pressure drop also increased with increasing liquid and gas load. Corresponding to the results for the total liquid holdup, no increase indicates a loading region before flooding occurs.

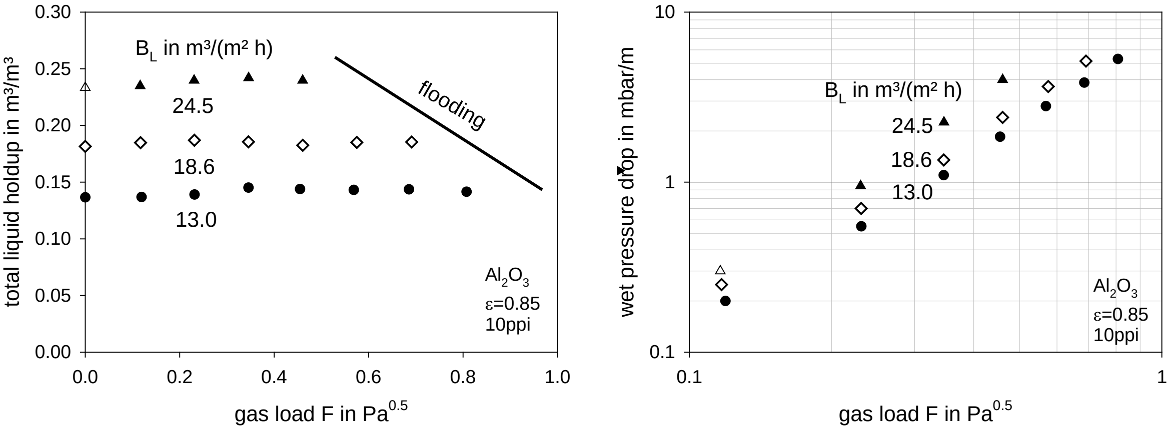

Figure 3. Total liquid holdup (left) and wet pressure drop (right) for one sponge type at different liquid loads B_L as a function of the gas load F.

A comparison of different sponge types at one liquid load is shown in Figure 4. The alumina samples with two different porosities show clearly the influence of porosity and sample quality. Samples with lower porosity and more closed faces lead to higher holdup values, higher pressure drops and flooding at lower gas loads. For the samples made of silicon infiltrated silicon carbide, lower holdup and pressure drop values were observed according to the static liquid holdup. Here the samples with higher pore number lead to flooding at lower gas loads but exhibit approximately the same total holdup values as the samples with lower pore number. For the samples with 10 ppi a loading region could be observed in holdup and pressure drop values. Thus loading was only observed for samples with high porosities, big pore sizes and a thoroughly opened structure. For the other samples, the retention of liquid due to a higher gas flow lead to immediate flooding and the formation of a liquid bubble bed on top of the packing, which was increasing in height up to 30 cm and lead to a significant liquid loss with the gas outlet. It was thus no longer possible to continue with the experiment. The description and comparison of the measured data with correlations from literature is topic of current work. It will be published in a forthcoming paper.

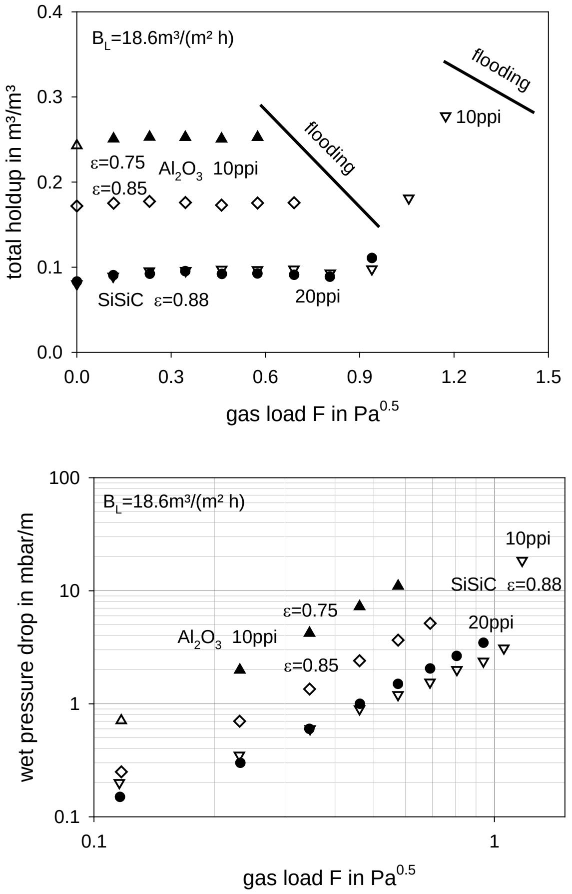

Figure 4. Total liquid holdup (left) and wet pressure drop (right) for different sponge types at a constant liquid load B_L as a function of the gas load F.

3.3 Mass transfer

The experimental determination of the gas and liquid side mass transfer coefficient as well as the effective surface area is topic of current research. Here only first results for the sponges made of silicon infiltrated silicon carbide with a pore number of 10ppi at different liquid loads are given in figure 5. It can be observed, that the effective interfacial surface area is lower than the geometric surface area at the investigated liquid loads (compare table 1). An increase of the effective surface area can be observed with increasing liquid and gas load. For the low liquid loads it is clearly visible that the surface area of the sponges is not properly wetted. Therefore very low effective surface areas are measured. Measurements for higher liquid loads are currently carried out and will be published in a forthcoming paper.

The mass transfer coefficients measured for the sponges made of silicon infiltrated silicon carbide with 10 ppi are in range of approximately $\beta_G = 5x10^{-3}$ m/s for the gas side and $\beta_L = 1x10^{-4}$ m/s for the liquid side determined from HTU_{OL} or $\beta_L = 5x10^{-5}$ m/s determined from HTU_{OG}. These values are in the range expected for packing structures concerning the liquid side, when compared to data of Hoffmann et al,[14], who determined the liquid side mass transfer coefficients from HTU_{OG}. As this data is less reproducible due to limitations in the determination of the gas-side concentrations in our experimental setup, the values determined from HTU_{OL} should be the reliable ones. For the gas side, the values found are significantly lower than expected, as Hoffmann et al.[14] determined values of approximately $\beta_G = 7x10^{-2}$ m/s. The values for gas and liquid side mass transfer coefficients fluctuate with the gas load, but no trend was visible in the first results. There was no clear dependence on the liquid load visible either. Results that will be obtained for other sponge samples have to be considered before a concise interpretation of these results will be possible.

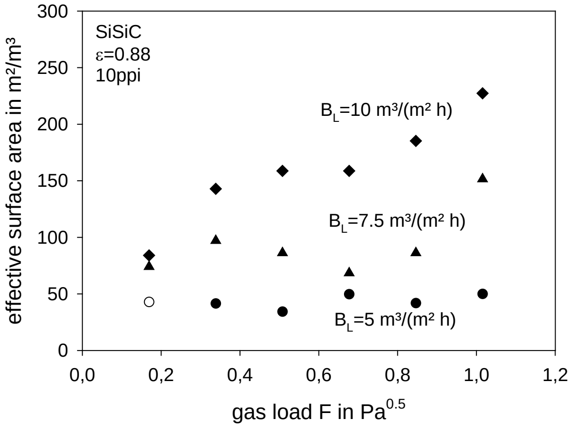

Figure 5. Effective surface area at different liquid loads B_L as a function of the gas load F for sponge samples made of silicon infiltrated silicon carbide.

4. Conclusions

Ceramic sponges exhibit comparably high static and total liquid holdup values. The onset of flooding often occurs without previous loading of the structure. Only selected sponge samples of good quality behave as would be expected for column packings. The onset of flooding occurs at comparably low gas load values, which limits the operation range. First results for the gas side mass transfer coefficients indicate lower values than could be expected for packing structures, for the liquid side they are slightly bigger. The measured interfacial area is low compared with the geometrical surface area of the samples. In general sponges qualify for applications in thermal process engineering, although they will be used preferably where inert material and the low pressure drop are of high importance.

Acknowledgements

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