**Micro-computed tomography for structural analysis of heterogenous ion-exchange membranes.**

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**Highlights**

* Micro-CT for structural analysis of ion-exchange membranes in their swollen state.
* Membrane swelling caused by the resin, shrinkage with increasing concentration.
* Method allows determination of ion-exchange area of heterogenous membranes.
* Sand equation shown not to hold for small pieces of the membranes.

**1. Introduction**

Electromembrane separation processes based on the use of ion-exchange membranes (IEM) play a very important role in production of potable water and increasingly in areas of waste water treatments. It is known that the performance of the whole process largely depends on the quality of the IEMs. These membranes can be classified into two major groups: (i) homogeneous, and (ii) heterogeneous. While homogenous generally posses better electrochemical properties they suffer from low mechanical and chemical stability. In these two aspects, they are outperformed by heterogenous ones which is however, at the expense of electrochemical performance. One of the ways how to improve the properties of heterogenous IEMs is too understand the relation between their structure and the exhibited behavior and later design membranes with optimal properties for a given application. This puts a requirement on having available analytical techniques that would allow to reconstruct 3D structure of these membranes and describe its composition quantitatively and qualitatively. At the same time these analyzed membranes should be practically tested regarding their performance.

To address the aforementioned problem we developed a technique which reconstructs 3D structure of membranes by micro-computed tomography and quantifies the composition not only in terms of volumetric composition but also in terms of surface composition which in turn provides very important data on the available ion-exchange area. Our method can work with both dry and wet (fully swollen) membranes which also allows one to study structural changes of membranes associated with swelling. We test our approach on testing the suitability of the Sand equation for determination of the fraction of conductive (ion-exchange) domains by combining our analytical technique with electrochemical measurement performed in a specific fluidic cell.

**2. Methods**

We developed a specific cell which allows microCT scanning of the samples in both dry and wet state. The details of this technique along with description of the evaluating methods can be found in [1]. Later the same piece of analyzed membrane can be integrated within a fluidic chip that allows electrochemical characterization. In this work, we measured so called transition times which are later used in the Sand equation to evaluate fraction of conductive domains. The details can be found in [2].

**3. Results and discussion**

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| a)  Figure 1. MicroCT surface analysis of a heterogeneous cation-exchange membrane. | Figure 2. Measurement of the transition times in the dependence of the applied electric current. |

Figure 1 and 2 show respective results of our studies focused on estimation of the ion-exchange area with two methods. The first one is based on the analytical reconstruction of the surface area by micro-CT and the other one on electrochemical measurement of transition times and substitution into the Sand equation. While microCT analysis is a direct technique that can locate the ion exchange-resin particles responsible for the actual ion-exchange (yellow color in Figure 1), the measurement of transition times is indirect technique that relies on several simplifications. Our results show that the electrochemical technique, although it might provide reasonable numbers, does not agree with results from microCT (compare red lines with black ones in Figure 2). Unlike that the Sand equation works very well for ion-exchange resin particles which can be considered as homogeneous and satisfies conditions under which Sand equation works.

**4. Conclusions**

Our technique based on micro-computed tomography allows to analyze the volumetric and surface structure of the heterogenous ion-exchange membrane, which are essential in electromembrane separation processes. By using this technique, we are able to characterize studied membranes in terms of composition and geometry. When combined with electrochemical measurements, we can relate the behavior of the membranes to the exhibited behavior.

**References**

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2. M. Svoboda, J. Beneš, L. Vobecká, Z. Slouka, J. Mem. Sci., 2017. 525: p. 195-201.