Ion exchange in continuous-flow system as an improved and efficient method for synthesis of ionic liquids

1. State the objective of the project.

The purpose of the project is conducting scientific research in order to develop a new and efficient methods for the synthesis of ionic liquids through the ion exchange reactions carried out in a continuous flow conditions and development of standard protocol for the synthesis of ionic liquids. Ion exchange reactions will be optimized for flow rate of liquid phase and choice both for solid and liquid phase. Using ion chromatography, the purity of the resulting ionic liquids, will be determined and compared with the purity of ionic liquids obtained by conventional methods (custom synthesis and industry standards). In addition, the physicochemical properties such as viscosity, melting point, thermal stability or water content will be determined and compared.

2. Research project methodology.

The research goal will be realized through the following research objectives: (1) synthesis of ionic liquids - optimization of reaction conditions, (2) examination of purity of obtained ionic liquids using ion chromatography (IC), (3) determination of the physicochemical properties of the obtained ionic liquids. The first step in the synthesis of selected ionic liquids will be the quaternization reaction between the selected cations to obtain the ILs with long alkyl chain substituents. Then, the obtained compounds will undergo an ion exchange reaction in column with filling. In the proposed project will be planned obtain the piperidinium and pyrrolidinium based ILs and ion exchange reaction on $[BF_4]^-$, $[NTf_2]^-$ i $[PF_6]^-$ anions. The obtained ionic liquids will complement a number of already known shorter alkyl chain ionic liquids based on those cations. It is important to determine the purity of the obtained ionic liquids.



Figure 1. Scheme of the continuous flow system.

All of the analyzes (viscosity, melting point, thermal stability or determination of water content, IR and ¹H NMR spectroscopy) will be performed to confirming that the new ILs have been synthesized and their basic physicochemical properties and the purity of ionic liquids will be compared with the properties of ionic liquids obtained by standard method.

3. Reasons for choosing the research topic.

Each of the available synthesis methods has limitations in terms of cations/anions selection, reaction conditions, or subsequent purification of the obtained ionic liquid. Therefore, conducting an ion exchange reactions in a continuous flow system will improve and modernize the already known synthesis methods. Currently, ILs synthesis methods only apply to those compounds with specific applications, while there is still a lacks of both the general procedures of synthesis and purification of the obtained ionic liquids. Dynamic development of this field, forces more and more new synthesis of ionic liquids with a specific structure and the specific properties (depending on the choice of cations and ions). In the literature, we cannot find any information about research on new methods of synthesis of ionic liquids by ion exchange in a continuous flow conditions using a packed column. This is an interesting alternative to the commonly used ion exchange reactions, which are performed in separatory funnels using for example methylene chloride(acetone)/water solvent biphasic systems. The use of the continuous flow conditions will allow the recycle and reuse of methylene chloride and regeneration of the column and its reuse. As a result of the performed research we will optimize proposed reactions in the respect to the flow rate of reactants and solvent to obtain ionic liquids with high purity and high yield.