Comparing synthetic methods to produce the ionic fluid 1-Pentyl-3-methylimidazolium tetrafluoroborate

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Summary -
During our project, we hope to synthesise the straight chain imidazolium into 1–pentyl-3-methylimidazolium tetrafluoroborate using two different methods, in order to compare the efficiency from an environmental perspective. We will change the solvent in each case to compare water and dichloromethane. Dichloromethane quickly evaporates to a gas, if it is released as a liquid. It will then decompose by reacting in the air with photochemically produced products. Dichloromethane has low acute toxicity to aquatic organisms, however there is not enough information to predict the long term effect that dichloromethane has on land animals and birds. Dichloromethane is not expected to bioaccumulate, however with a lack of temporal evidence this cannot be confirmed for certain. It is not expected to react with ozone in the upper atmosphere since most of it will decompose in the lower atmosphere, but we anticipate that this will give a higher yield. So far we have attempted the water method that has consistently produced an oily substance, that we will analyse at Imperial College London and use this time to complete the second part of our project, as our school lab limits us.

Aims -
To compare two methods using different solvents (water and dichloromethane) to make the ionic liquid 1–Pentyl-3-methylimidazolium tetrafluoroborate, and compare how ecologically friendly they are. The use of dichloromethane has drawbacks particularly compared to water. These drawbacks include: affecting the central nervous system of the human body, slight exposure to dichloromethane can cause hearing and vision impairments, direct contact with human skin causes redness of the skin along with intense burning. Therefore, we will evaluate the yield of both methods to see if the increased yield outways the environmental impact of using dichloromethane.

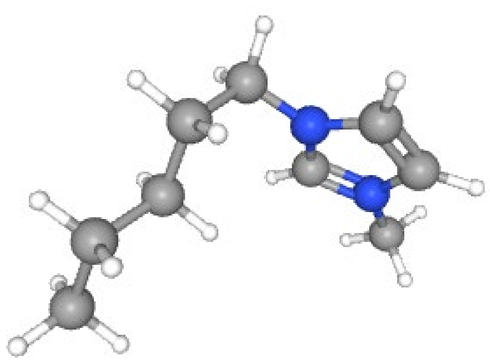

Figure 2 - 1 - pentyl, 3-methylimidazolium tetrafluoroborate.https://pubchem.ncbi.nlm.nih.gov/

Results -
So far we have only run the test with water as a solvent, as in our school lab we do not have access to a rotary evaporator, meaning that we can't use dichloromethane as a solvent. We have found that by increasing the time we reflux by a few hours, we have significantly increased the yield produced by the reaction. This has produced a small amount of oily product, which floats around in the solvent and is largely viscous, as well as another product, in the form of white crystals which sit on the bottom of the substance showing that it has a higher density than the substance within it. We expect that our ionic liquid (1–Pentyl-3-methylimidazolium tetrafluoroborate) is the oily layer, and we intend to analyse our product using NMR at Imperial College London next week to allow us to take our research further. When we use dichloromethane as a solvent, we expect to have a significantly higher yield than that produced by water.

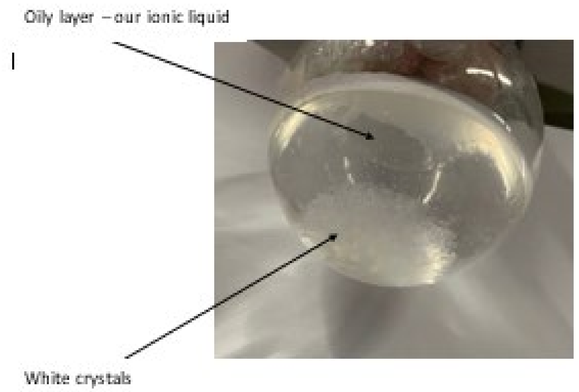

Figure 1: white crystals and oily product post reflux

Method - water solvent

1. Add 20g of deionised water, 3.74g of methylimidazole, 7.01g of bromopentane and a few anti-bumping granules into a 250ml round bottomed flask.
2. Heat under reflux for 2 hours.
3. Add 5.8g of potassium tetrafluoroborate straight into the round bottomed flask. Continue to reflux for 45 mins.
4. Allow to cool for 10 mins
5. 8) To separate the 2 layers, pour the mixture into a separating funnel, remove the bottom layer into a clean container.

Method - Dichloromethane as a solvent

1) Add dichloromethane, 3.74g of methylimidazole, 7.01g of bromopentane and a few anti-bumping granules to a 250ml round bottomed flask.
2) Place the round bottomed flask to a heating mantle.
3) Set up reflux condenser using clips and lubricants.
4) Connect slow running water through the bottom pipe.
5) Heat under reflux for 2 hours.
6) Remove from reflux and add 5.8g of potassium tetrafluoroborate straight into the round bottomed flask. Continue to reflux for 45 mins.
7) Take off of reflux and leave to stand for 10 mins
8) To separate the 2 layers, use a rotary evaporator to remove the dichloromethane and leave a dry product.

Next steps -
We will go on to compare theoretical yield, atom economy, eFactor, boiling points and ecological impact / efficiency of each method so we are able to produce the most economic product. This evaluation will go on to decide what method we will use to create 1 –Pentyl-3-methylimidazolium tetrafluoroborate. This is because if by using water as a solvent, which is more ecologically friendly, we produce a smaller yield, then we will need to weigh up whether the benefits and potential applications of our product will outweigh the disadvantage of the ecological impact of producing the ionic liquid in the first place.

References

https://pubchem.ncbi.nlm.nih.gov/

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