Greener Fragrances: Making Ionic Liquids Iconic

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Summary

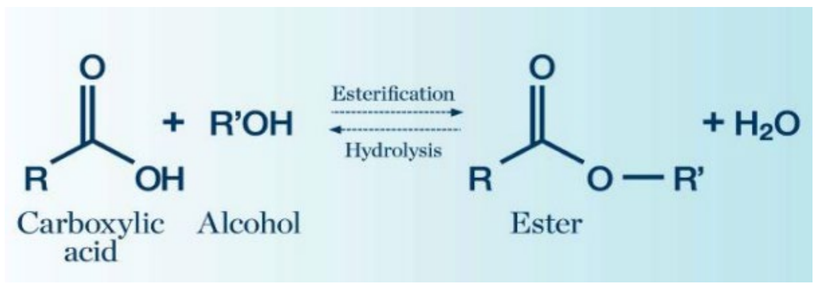
Traditional metallic compounds are commonly used as catalysts in organic reactions. These resources are becoming increasingly difficult to extract due to finite resources running out. These reactions also typically require vast amounts of energy due to reactions requiring high temperatures. This project aims to find a new catalyst, an ionic liquid. These ionic liquids can catalyse reactions at lower temperatures, thus reducing the amount of fossil fuels that must be used to generate energy to heat these reactions.

Background Information

Esterification reactions are reactions between carboxylic acids and alcohols to produce esters. This reaction is typically slow and requires heat and a concentrated strong acid (normally sulfuric acid) to react. These esters are in high demand in the fragrance industry, meaning that vast amounts of harmful acid catalysts are used. These acids then have to be extensively processed to ensure they are pure for another esterification reaction. This project aims to turn away from acid catalysts by producing protic ionic liquids which can be used to catalyse the reactions instead.

Research Aims

- The successful synthesis of Triethylammonium Hydrogen Sulfate [TEA][HSO4]
- To produce an ester avoiding the use of sulfuric acid as a catalyst
- To analyse the esters we produced to see if there was a difference in yields when using different ratios of acid to base when producing the protic ionic liquid



Esterification reactions
This shows a carboxylic acid reacting with an alcohol to produce an ester and water.

Experimental Method to Produce a Protic Ionic Liquid

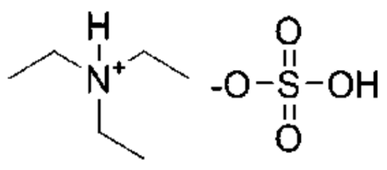
We planned to produce two slightly different ionic liquids. We were going to produce one with a 2:1 acid to base ratio, and a 3:1 acid to base ratio. We were then going to attempt to produce ethyl ethanoate and butyl salicylate. We were then going to send our samples away to be tested by NMR analysis to determine which ratio of acid to base in the ionic liquid produced the highest yield of ester.

1. The appropriate mass of triethylamine was weighed out in a beaker and transferred to a round bottom flask
2. The flask was then placed in an ice bath and a stir bar was added
3. the sulfuric acid should be weighed out in a separate beaker.
4. The stirring should be turned on, and the sulfuric should have been added dropwise
5. The solution should then be left stirring vigorously for 1 hour, then should be removed from the ice bath and stirred for a further 3 hours at room temperature

Reaction taking place:

$$N(C_2H_5)_3 + H_2SO_4 \rightarrow [N(C_2H_5)_3]^+ + [HSO_4]^-$$

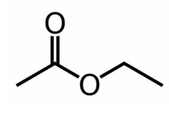
The sulfuric acid being added to an ice bath
This was to prevent the highly exothermic reaction from heating up, as triethylamine can spontaneously ignite at relatively low temperatures



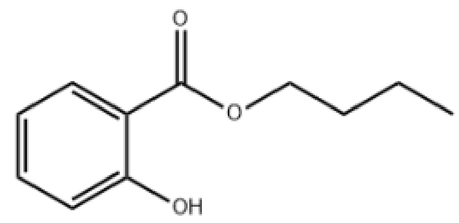
Formula of triethylammonium sulfate
Above is the chemical formula of the protic ionic liquid we produced

Experimental Esterification Method

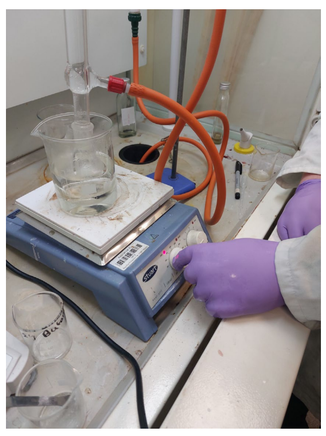
- Measure out the appropriate volume of ethanoic acid in a beaker, and transfer it into a round bottom flask
- Add the appropriate volume of ethanol along with 1.5%mol of the 2:1 ionic liquid.
- This should then be heated to 90°C for 30 minutes being refluxed.
- Once refluxed, the solution should then be transferred to a separating funnel, where two clear layers should form
- The denser ionic liquid should settle at the bottom of the flask and should be filtered off. The ethyl ethanoate should then be filtered into a glass storage container, sealed, and labelled.
- This should then be repeated with the 3:1 ionic liquid
- All of this should then be completed using salicylic acid and butanol.



Ethyl ethanoate



Butyl salicylate



Ester synthesis – reflux setup
The image above shows our heating plate and string, along with our reflux condenser and our round bottom flask, being heated to 90°C for 30 minutes

Our Samples
From left to right:
2:1 ethyl ethanoate, 2:1 used ionic liquid, 3:1 ethyl ethanoate, 3:1 ethyl ethanoate, 3:1 used ionic liquid,

Challenges we Encountered

- Our first problem occurred when refluxing the ethyl ethanoate. The glassware was not completely sealed which resulted in us losing a large volume of ester from the reaction.
- Our second problem was during the separation of ethyl ethanoate. We were using very small volumes of liquids which made it difficult to distinguish between layers in the separating funnel. To avoid this in the future, we tripled our volumes when producing the butyl salicylate esters.
- Our third problem was due to an issue with solubility that led to an excess of salicylic acid being added to the solution, which did not dissolve, preventing stirring, and making it difficult to filter any remaining ester that was suspended in the salicylic acid
- Our fourth problem was that by the time this poster is submitted, we won't have sent our samples off for NMR analysis, but we should still have some data with us at the conference.

Our Predictions

We are predicting that the esters produced using the 3:1 ratio of sulfuric acid to base ratio will produce the greatest yields of esters, but we believe that due to inaccuracies in the filtration process, some of the esters will have been lost, and some of the ionic liquid catalyst will have remained in the solution, which could produce impurities during testing

Acknowledgments

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