Influence of pH and acid type on the reaction of acetaldehyde and ammonia

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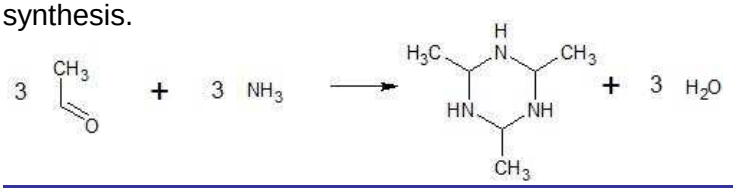
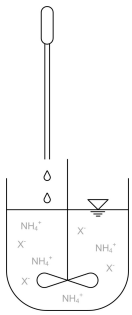
This study deals with the screening of reaction conditions for acetaldehyde ammonia trimer synthesis, testing the reaction in the entire spectrum of pH, from highly basic to highly acid, thereby investigating 3 different acid types.

Acetaldehyde ammonia trimer

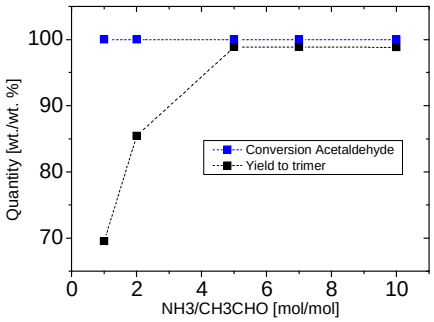
Acetaldehyde ammonia trimer is a cyclic molecule produced from reaction of acetaldehyde and ammonia. It is used as a raw material in organic synthesis.





Experimental setup

- Continuously stirred and cooled reactor
- Reaction at RT and atmospheric pressure
- Slow addition of acetaldehyde to ammonium salt solution in 1:1 stoichiometric ratio

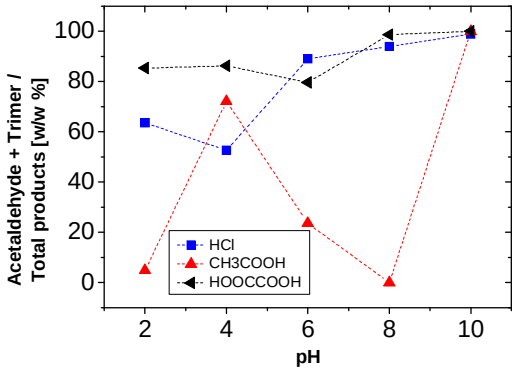


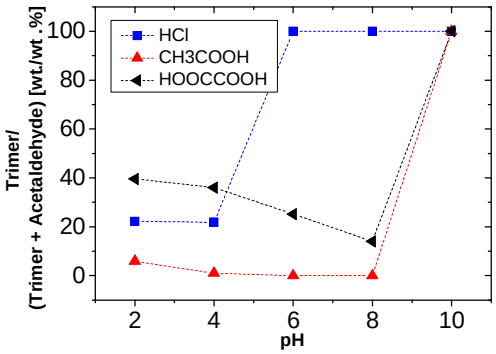
Trimer synthesis

- Reference: acetaldehyde addition to 25 % ammonia solution in water, pH = 14
- Efficiency in trimer production strongly depends from stoichiometric ratio of ammonia on acetaldehyde.
- Full production of trimer is possible only at ratio > 5

Influence of pH on trimer formation

- General decrease of acetaldehyde and trimer with decreasing pH
- Hydrochloric acid shows a steep decrease in trimer and ammonia recovery from pH 6 to pH 4, from ~ 90 % to ~ 60 %
- In acetic acid acetaldehyde is completely decomposed at pH 8 and pH 2. A relative maximum is visible at pH 4
- Differently, oxalic acid stabilize acetaldehyde and trimer.





- Relative ratio acetaldehyde-trimer strongly depends on acid type
- Hydrochloric acid shows a drop in trimer selectivity with decreasing pH from pH 6 to pH 4, stabilizing at lower pH at 20 % of trimer
- In acetic acid no trimer is formed at pH lower than 10
- The trend is slightly different in oxalic acid. A minimum is found at pH 8, because increase of acidity favours trimer formation

Conclusions

It is possible to optimize the mixture of ammonia and acetaldehyde in order to minimize polymer production and acetaldehyde loss. Nevertheless it is difficult to achieve high yield of trimer at pH other than basic. The acid used has an important effect, since it can stabilize acetaldehyde or catalyse polymerisation, modifying yield and selectivity.

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