Friedrich-Alexander-Universität Erlangen-Nürnberg

ENGINEERING OF ADVANCED MATERIALS

Catalysis in Low Temperature Molten Salts

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Low Temperature Molten Salts can bridge the gap between ionic liquids and classical high temperature molten salts

Ionic liquids
- Organic cations + anion
- T_m < 100 C
- T_{stable} < 250 C [1]

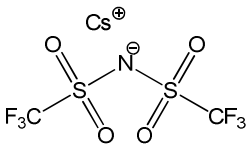
Low Temperature Molten Salts
- Inorganic cation + organic anion
- 100 C < T_m < 250 C
- T_{stable} > 250 C

High Temperature Molten Salts
- Inorganic cation + anion
- T_m > 400 C
- T_{stable} >> 400 C

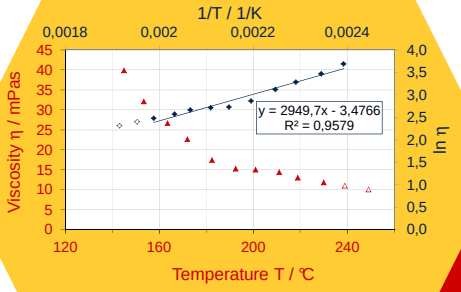
Promising candidate:
Alkali bis(trifluoromethylsulfonyl)amide M[NTf_2]
- High chemical and thermal stability [2], [3]
- Weakly coordinating counter-ion [4]

High Temperature Homogeneous Catalysis
- Dissolving catalyst precursors in the molten salt
- Application: endothermic processes
- Using solubility effects of educts and products for shifting the equilibrium

Physico-chemical properties of Cs[NTf_2]

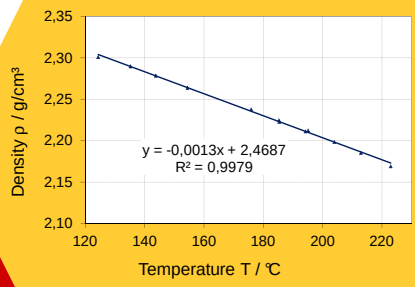


Viscosity



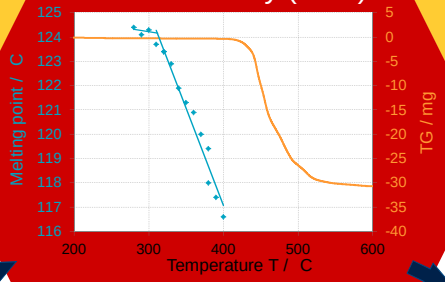
Exponential dependence on temperature
Newtonian behaviour

Density



Linear function of temperature

Melting point (DSC) and thermal stability (TGA)

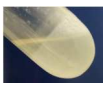


Results from DSC
Melting point depression from 300 C

Results from TG-Analysis
Mass loss from 400 C

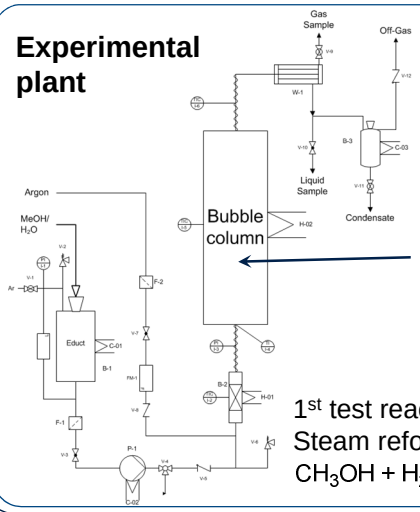

$CsNTf_2$ at 200 C


$CsNTf_2$ at 320 C for > 400 h


$CsNTf_2$ at 340 C

- Similar results like DSC visible change of molten salts at T > 320 C
- Beginning decomposition from 300 C but still salt like non volatile structure
- TG analysis not suitable for exact thermal stability analysis

Experimental plant



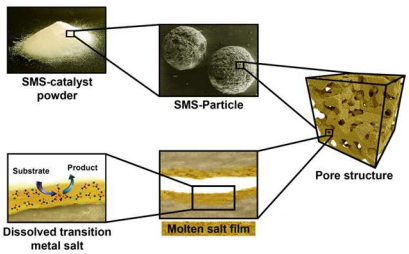
Bubble column reactor for testing molten salt-catalyst-systems at temperatures up to 400 C and atmospheric pressure

Liquid stationary phase:
- Molten salt + dissolved catalyst

Gas phase:
- Educts + products

1st test reaction:
Steam reforming of methanol
$CH_3OH + H_2O \longrightarrow 3\,H_2 + CO_2$

Outlook



Developing Supported Molten Salt (SMS) Catalyst for High Temperature Homogeneous Catalysis

[1] S. Werner, M. Haumann, P. Wasserscheid, *Annu. Rev. Chem. Biomol. Eng.* **2010**, *1*, 203-230.
[2] R. Hagiwara, K. Tamaki, K. Kubota, T. Goto, T. Nohira, *J. Chem. Eng. Data* **2008,** *53,* 355–358.
[3] J. P. Leal, et al., *J. Phys. Chem. B* **2009**, *113,* 3491–3498.
[4] P. Wasserscheid, P.S. Schulz in P. Wasserscheid, T. Welton (eds.), *Ionic Liquids in Synthesis*, 2nd edition, „Transition metal catalysis in ionic liquids", Wiley-VCH, Weinheim, **2007**, 369-463.

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