METHODOLOGY OF SAFETY DEVELOPMENT FOR HYDROGENATION PROCESS

Domagoj Prlić, Ivan Vrban, Igor Kultan, Ozren Wittine, Eugen Marcelić

PLIVA Hrvatska d.o.o., Prilaz baruna Filipovića 25, 10000 Zagreb

E-mail: domagoj.prlic@pliva.com

INTRODUCTION :

Reaction calorimetry measures the heat released from a chemical reaction or physical process and provides the fundamentals of the thermochemistry and kinetics of a reaction. The information obtained is essential to describe the heat release of a chemical reaction over time, which can uncover unexpected behavior and make any scalability issues visible and quantifiable. Measured data are used to characterize, optimize and understand process parameters in a controlled, accurate and reproducible environment, and to enable safe scale-up and transfer into manufacturing.

HYDROGENATION:

Hydrogenation process is usually used to reduce or saturate organic compounds. Catalysts are needed for the reaction to be usable; noncatalytic hydrogenation takes place only at very high temperatures.

EXPERIMENTAL PART :

During experimental work RC1 and ARSST were used to determine the reaction enthalpy generated by a chemical reaction (Figure 1.). Compound X is prepared by reduction of nitro compound Y. The reduction is performed as catalytic hydrogenation in MeOH as solvent and with 5% Pd/C as catalyst. Reaction temperature is 45 °C and pressure of hydrogen is 5 bar.

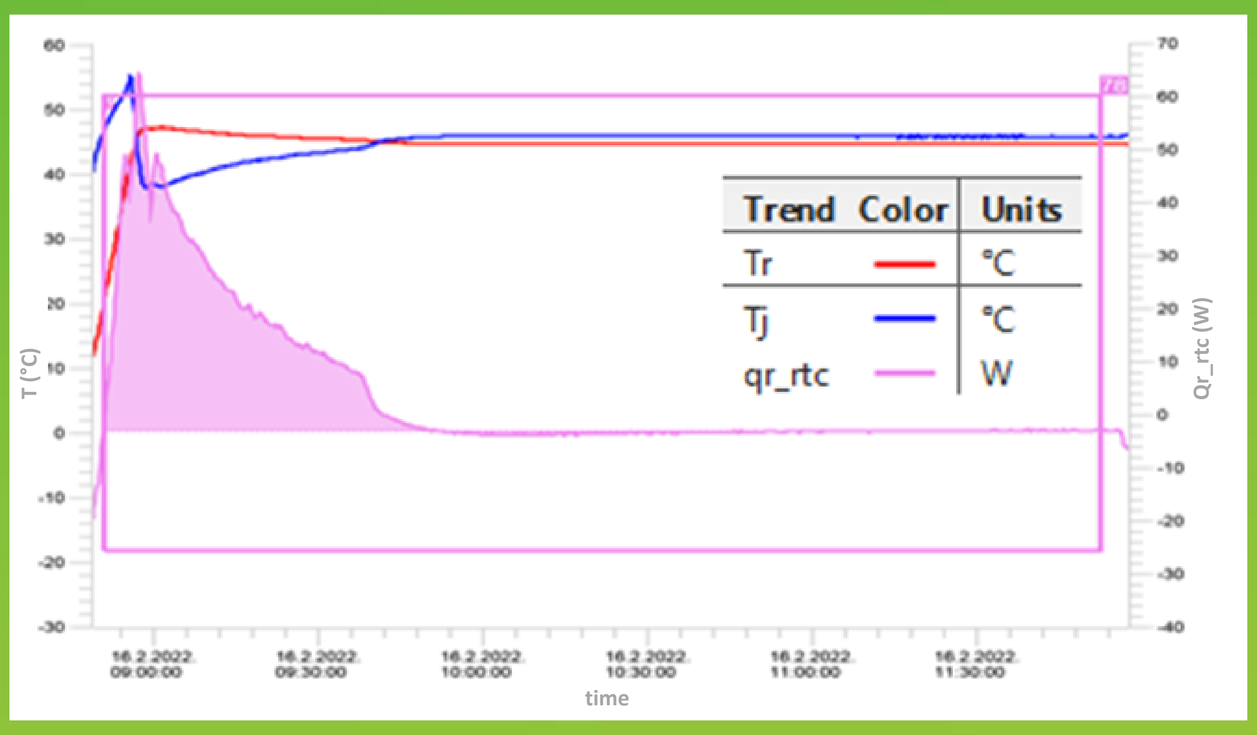

Figure 1. Enthalpy generated by a chemical reaction measured in RC1

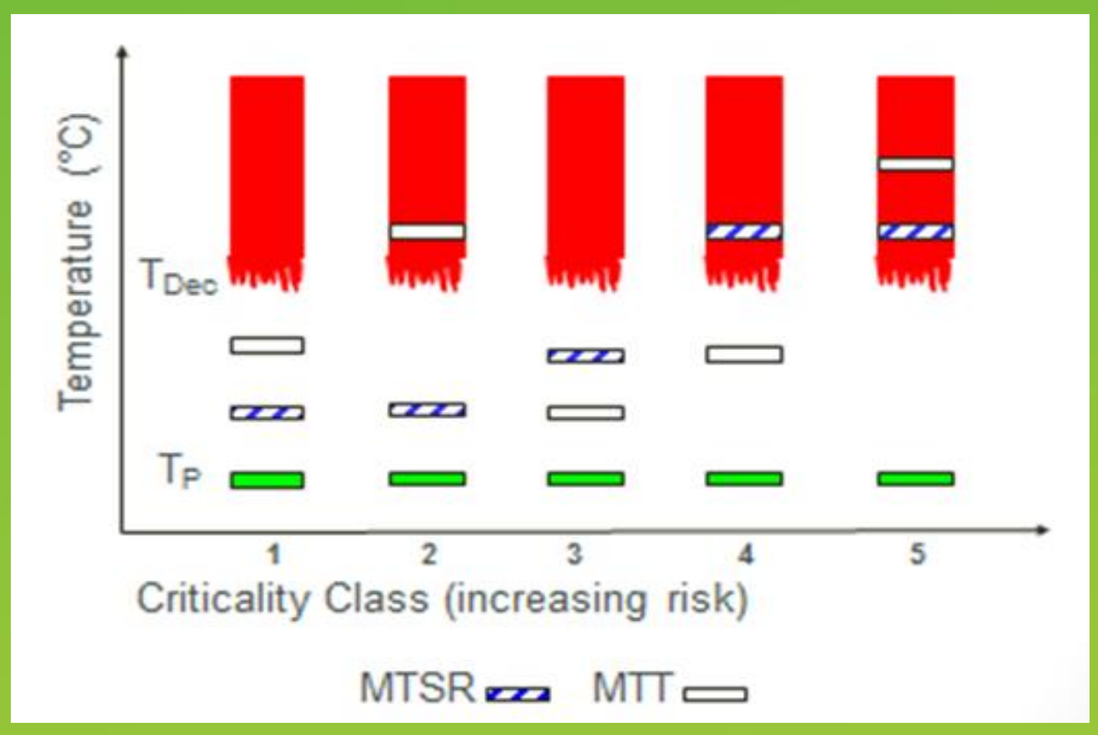

Figure 2. Criticality classes of scenario (Stoessel, 2009).

CONCLUSION:

Based on calorimetric and thermodynamic data which are measured, criticality classes are determined and due to MTSR (128°C) being higher then MTT (65°C) reaction belongs to criticality class 3. According to the DSC (Differential scanning calorimetry) data, material is thermally sensitive because of low onset temperature (95 °C), but with very low decomposition energy. Because of these data, Yoshida's correlation is used and results for SS (Shock sensitivity) and EP (Explosion propagation) are negative. The reaction enthalpy generated by the chemical reaction is 78.041 kJ.

MTSR = 128 °C
(Maximum temperature of synthesis reaction)

MTT = 65 °C
(Maximum temperature of synthesis reaction)

T_{dec} > 120 (per ARSST)

T_{dec} = 217 °C (per DSC)
(Temperature of decomposition)

T_p = 45 °C
(Process temperature)

međunarodni znanstveno-stručni skup
19. Ružičkini dani
DANAS ZNANOST – SUTRA INDUSTRIJA
21. – 23. rujna 2022. | Vukovar, Hrvatska