Sticking coefficient – Correlation between QTGA & CVCM

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07/11/2019

Outline

- Objective
- Information on the facility
- Measurements
- New modelling approach
- Conclusion

Some terminology recapping (ECSS-Q-TM-70-52A)

Quartz Crystal Microbalance(QCM) Thermogravimetric analysis (TGA): Measures weight/mass change(loss/gain) and the rate of weight change as a function of temperature, time and atmosphere.

TML : Total Mass Loss

RML : Recovered mass loss

CVCM : Collected volatile condensable material

KC: Knudsen Cell

LN2: Liquid Nitrogen

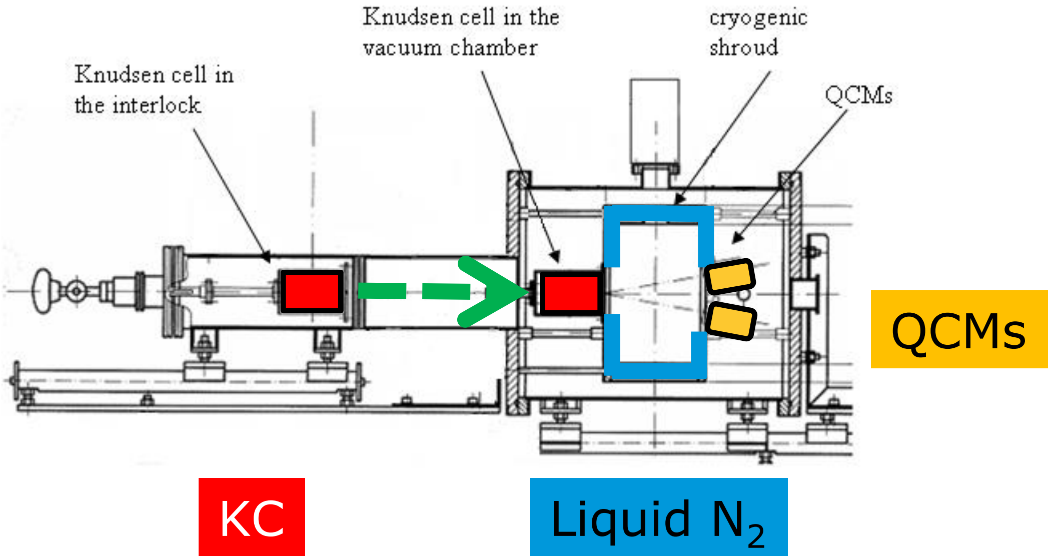
DOK: Dynamic outgassing Knudsen

Standard Test: TGA performed at the end of the test

EOT: End of Test

The DOK schematic & Multi-step temperature test method

Sample temperature is increased from 25°C to X°C by steps of 25°C every 24h. The number of steps and the duration at the max. test temperature is adjusted based on the real application temperature and the max. permitted test temperature of the sample.



$T_{max} \cong 450°C$

$\bar{T} \cong -175°C$

$P < 5 \times 10^{-6} mbar$

$T_{QCM,1} \cong -170°C$

$T_{QCM,2} \cong -25°C$

$T_{QCM,3} \cong -75°C$

$T_{QCM,4} \cong -50°C$

Input parameters generated by current approach

1) Mathematical species

Time constant $\tau_{0,i}$ (h)	Initial mass $W_{0,i}$ (%)
0.5	0.0058
4.482096	0
40.17836	0.13660
360.1665	0.14052
3228.602	0.04488
28941.81	0.00067
259439.9	0.00312
2325669	0.00356
20847740	0.01035

2) A table which contains activation energies and temperature-time constant

Period	Temperature (°C)	Acceleration Factor $K_{i \to i+1}$	Apparent Activation Energy $E_{i \to i+1}$ (kJ·mol[-1])	Residence time-temperature dependency coefficient k_e
I→II	75	4.12	52.9	0.0834
II→III	100	3.65	52.9	
III→IV	125	5.66	56.0	
IV→V	150	20.68	85.7	
V→VI	175	23.18	169.7	

3) TML, RML and CVCM of the sample

4) A clear description of the sample and the test methodology

5) TGA analysis which contains further information which may improve contamination modelling

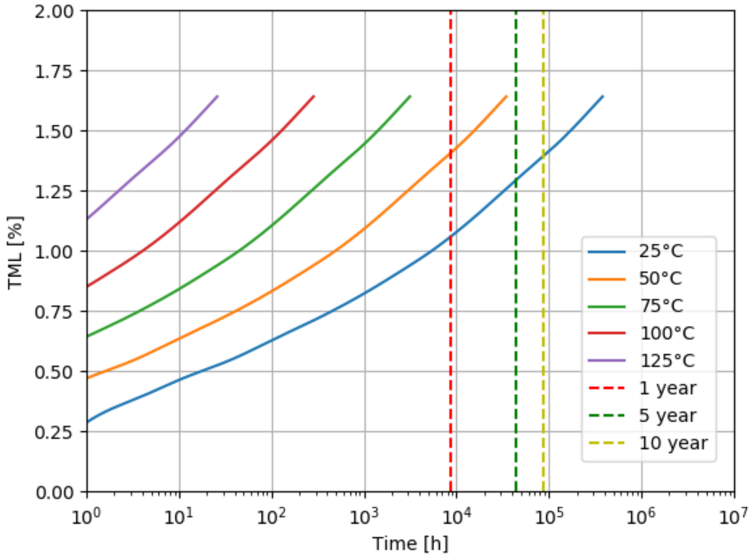
6) Reemission parameters are calculated by the assumption of $T_{ref} + 50K$

Long-term outgassing predictions

Long-term outgassing modelling is based on a step-wise dynamic outgassing test. The experimental data collected over few days is extrapolated to many year mission through a power law.

$$W(T) = \sum_i W_{0,i} \cdot (1 - e^{-t/\tau_{T,i}})$$

$$\tau_{T,i} = \tau_{0,i} \cdot e^{-k(T - T_{ref})}$$



W(T) – TML or CVCM

$\tau_{T,i}$ - time constant of a specie i at T

t - time in hours

T_{ref} - usually 25°C

k – residence time-temperature dependence coefficient (*k* emission and *k* reemission)

Objectives of this work

Currently, in Europe, there are multiple on-going studies to develop new tools and/or to implement new parameters into contamination modelling to increase the accuracy of the obtained results from simulations.

The state-of-art method to estimate reemission parameters is based on a crude approach: Assuming that reemission time constants are equal to the outgassing time constants at temperature $T + 50K$. This rule often significantly overestimates the experimental data.

This work is a first approach to improve the prediction accuracy from step-by-step TGA analysis run on a QCM.

The DOK schematic & dynamic QTGA approach

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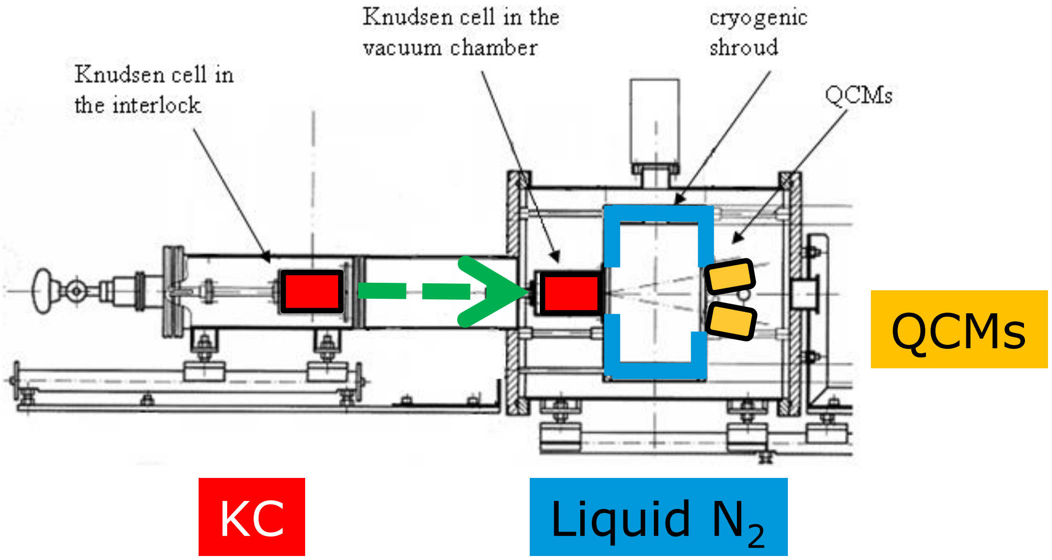

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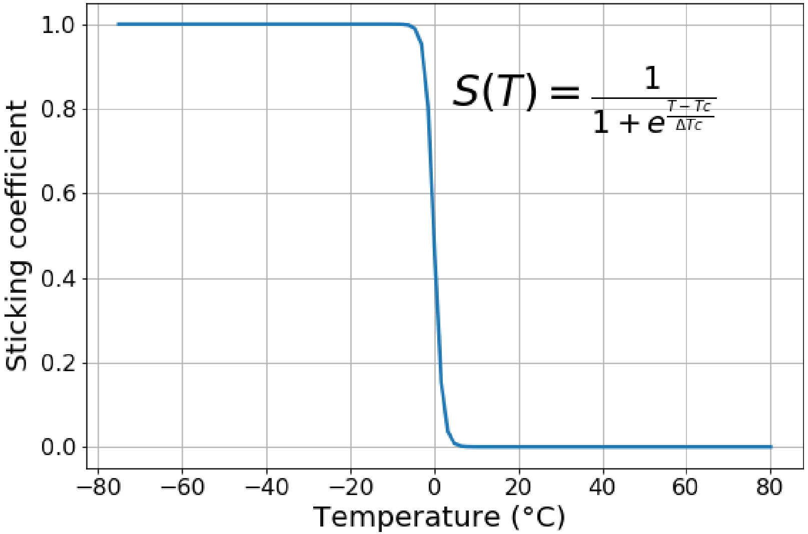
$P < 5 \times 10^{-6} mbar$

Standard test	*Comparison test*
$T_{QCM,1} \cong -170°C$	$T_{QCM,1} \cong -170°C$
$T_{QCM,2} \cong +25°C$	$T_{QCM,2} \cong -25°C$
$T_{QCM,3} \cong -75°C$	$T_{QCM,3} \cong -75°C$
$T_{QCM,4} \cong +50°C$	$T_{QCM,4} \cong -50°C$

Sticking coefficients

Logical function to define sticking in terms of surface temperature



$$m_{deposit}(T, T_{QCM}) = \sum_{\alpha} S_{\alpha} \cdot w_{\alpha} \cdot m_{CVCM(T_{QCM})}$$

S_{α} - sticking coefficient for species α

w_{α} - weight fraction of species α

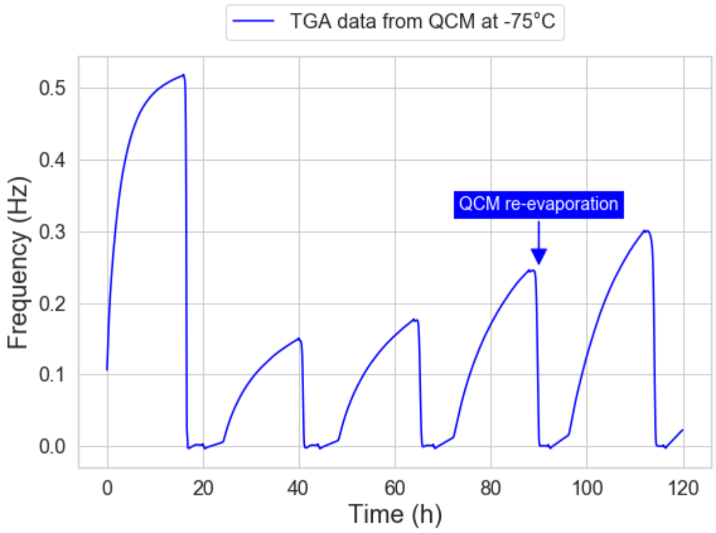
$m_{CVCM(T_{QCM})}$- total contaminant deposit on a QCM at a temperature (T_{QCM})

Tc_{α} - captation temperature for species α

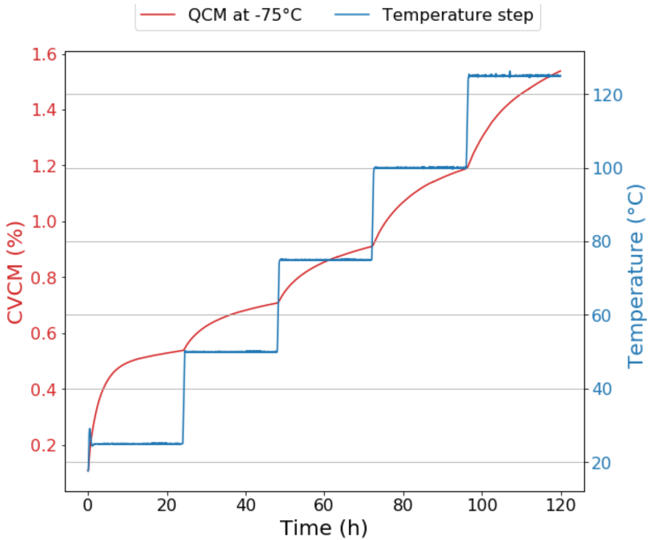
ΔTc_{α}- transition coefficient for species α

New testing approach - QTGA

Re-evaporation of a QCM at -75°C after each temperature step



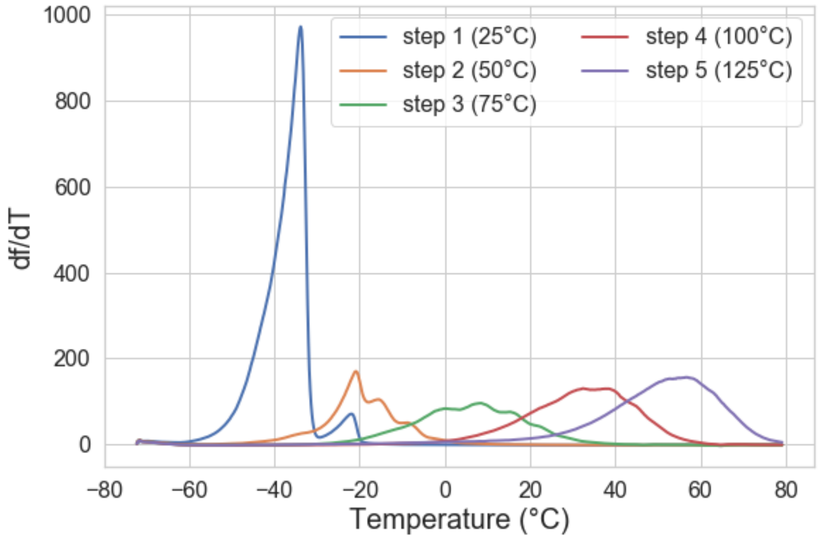
Raw data from a QCM at -75°C

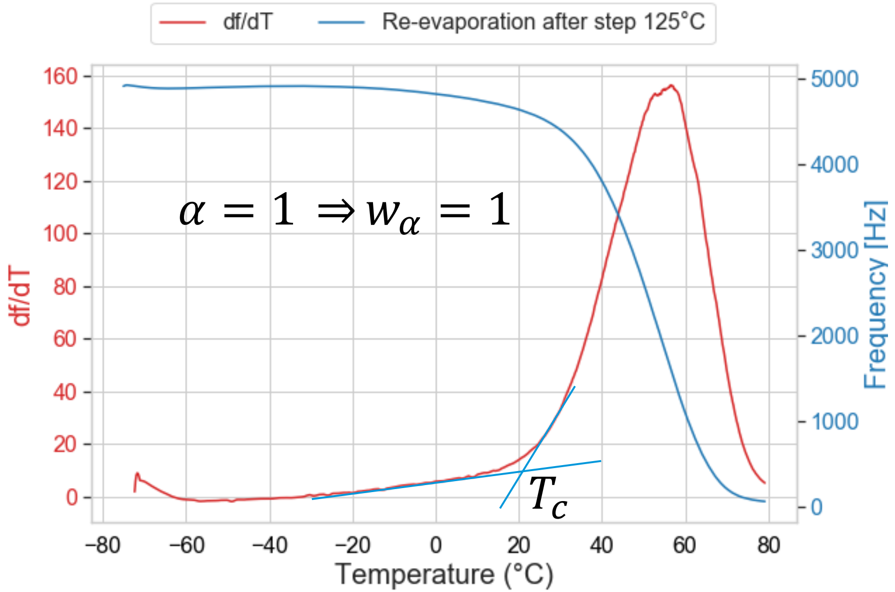


Data merged into a single CVCM curve

New testing approach - QTGA

TGA performed after each step shows the spectrum of chemical species

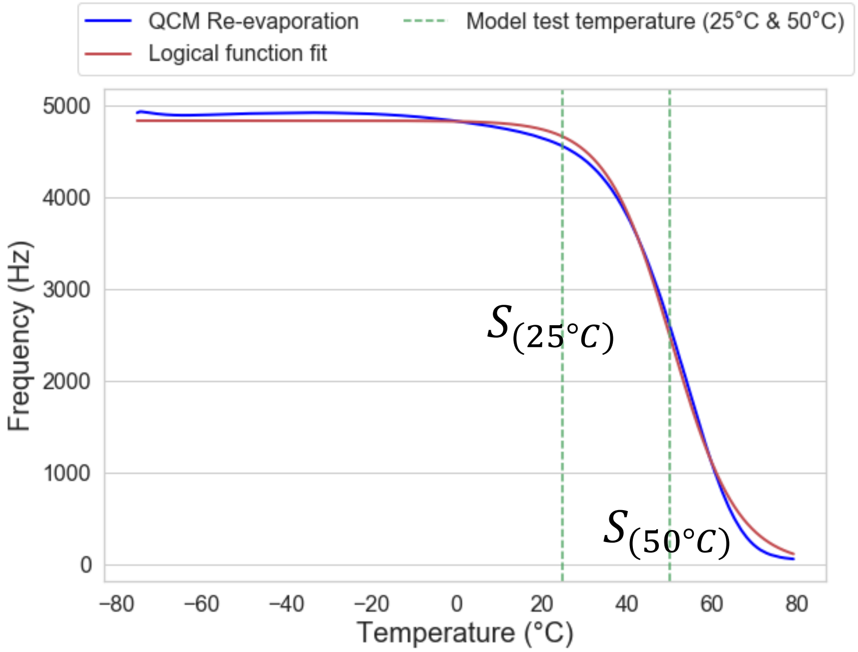


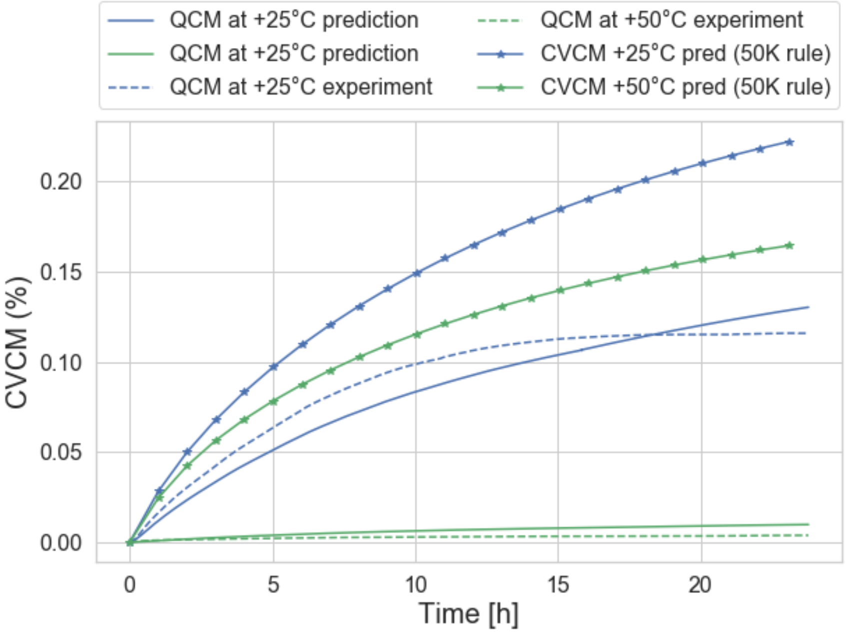


$$m_{deposit}(T, T_{QCM}) = \sum_\alpha S_\alpha \cdot \overbrace{w_\alpha}^{=1} \cdot m_{CVCM(T_{QCM})} = S_\alpha \cdot m_{CVCM(T_{QCM})}$$

Experiment vs. prediction

Material: RT 745 – typically single peak per temperature step (single function fit)

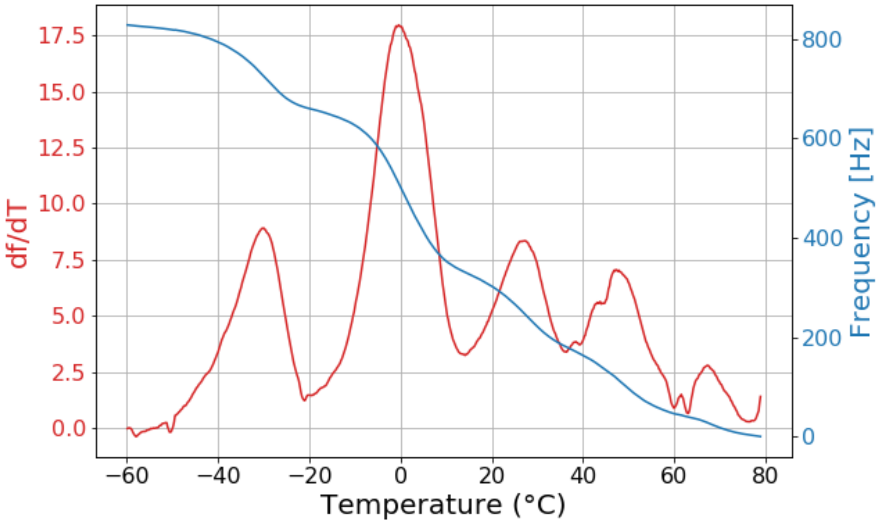


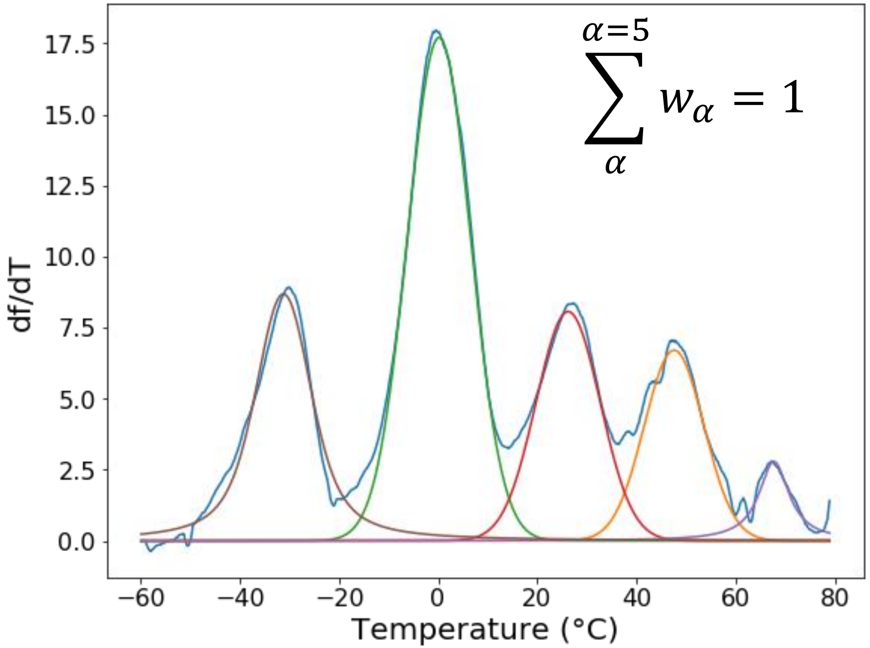


$$m_{deposit}(T, T_{QCM}) = S_{\alpha} \cdot m_{CVCM(T_{QCM})}$$

Experiment vs. prediction

Material: Kapton – typically multiple peaks per temperature step (function sum fit)



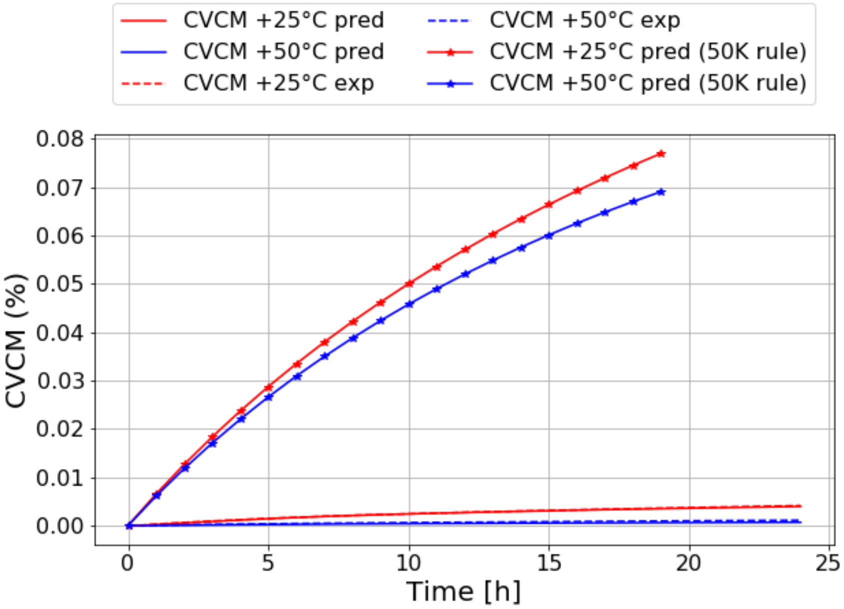


$$\sum_{\alpha}^{\alpha=5} w_\alpha = 1$$

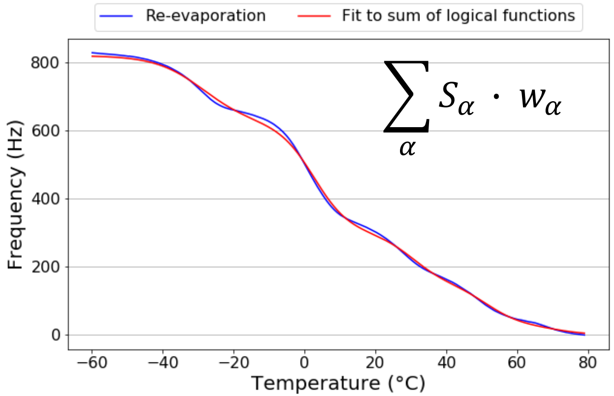
Multiple individual peaks are observed for each temperature step – peaks deconvolution aids in assigning weights to each peak.

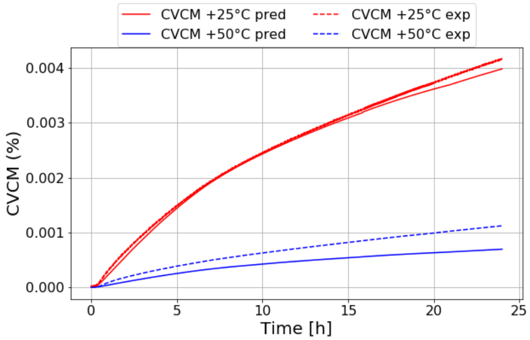
Experiment vs. prediction

Material: Kapton – typically multiple peaks per temperature step (function sum fit)



Zoom in





Improved modelling approach

Sticking coefficients

- Model is based on the physical phenomena
- Error is within few percent for a duration of 24hours.

+50K rule

- Model is based on mathematical fitting – which often produces non-physical results
- Model overestimates CVCM rates
- Error is large

Material	RT747				Kapton			
QCM temperature	+25°C		+50°C		+25°C		+50°C	
	CVCM (%)	Error (%)	CVCM (%)	Error (%)	CVCM (%)	Error (%)	CVCM (%)	Error (%)
Experiment	0.1158	-	0.0037	-	0.0041	-	0.0011	-
Prediction S(T)	0.1302	12%	0.0097	162%	0.0039	4.87%	0.0007	36%
Prediction +50K	0.2219	91%	0.1643	4340%	0.0770	1778%	0.0690	6172%

Conclusion

- The new modelling approach shows a potential to improve the current mathematical fitting model based on a physical phenomena.

- The future work will focus on:

  A) more specific definition of captation temperature

  B) including kinetic parameters in the model obtained from a TGA profile

  C) modification of a model to predict the CVCM data from a TGA profile

  D) Model validation with a range of materials