

# Calibration and validation of model describing complete autotrophic nitrogen removal in granular sludge

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# Calibration and validation of model describing complete autotrophic nitrogen removal in granular sludge

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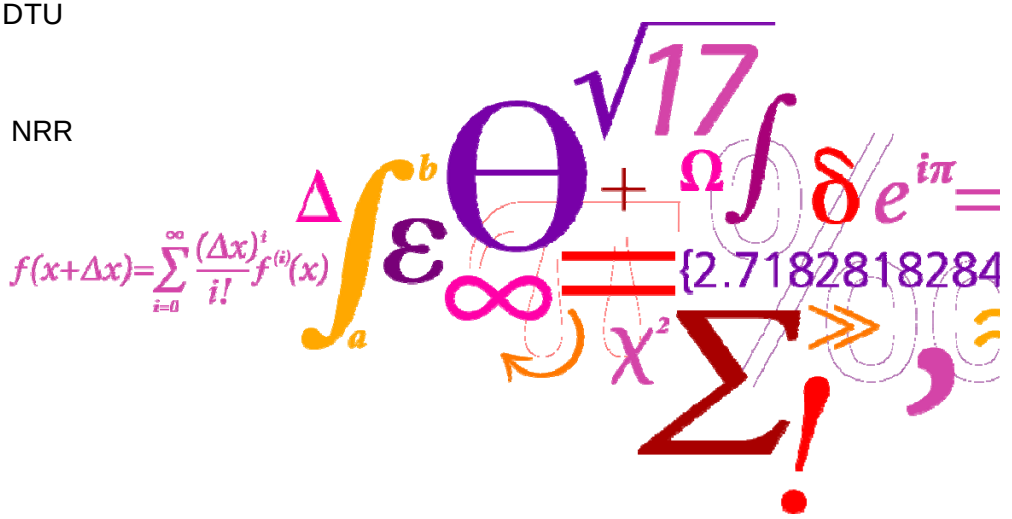


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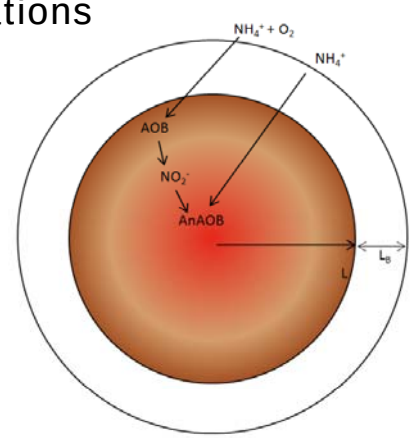
# The case

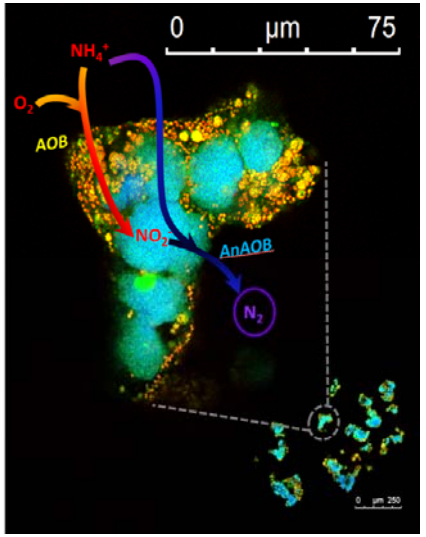
A granular sludge SBR performing N removal through nitritation/anammox

- Calibration methodology developed
- Fast model initialization
- Stoichiometric ratio evaluation

Purpose:

- Experiment planning
- Performance prediction for control applications



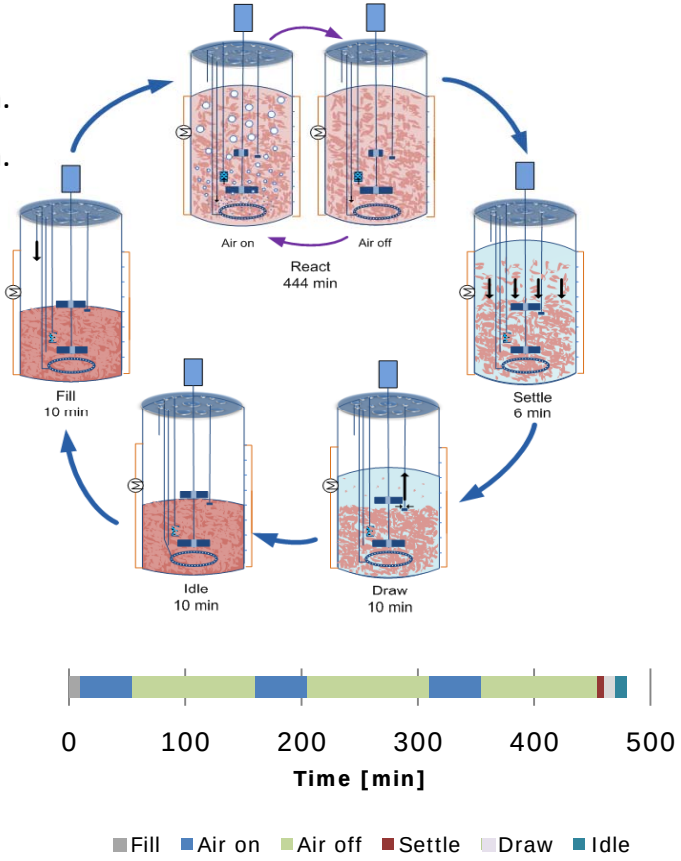
# Methods
# Physical system

**Reactor characteristics:**

| | |
|---|---|
| Volume: | 4L |
| Temperature: | 30°C |
| pH: | 7.5±0.3 |
| Mixing: | 6-bladed Rushton impeller at 80 rpm + bubble aeration |
| Solids concentration: | 4.2 g VSS/L |
| Ave. gran. size: | 50 µm |
| Operating time: | 11 months |

**Sequencing batch operation:**

| | |
|---|---|
| Fill: | 10 min. |
| Reaction: | 444 min. consisting of three aerated phases and three non-aerated phases |
| Settling: | 6 min. |
| Draw: | 10 min. |
| Idle: | 10 min. |

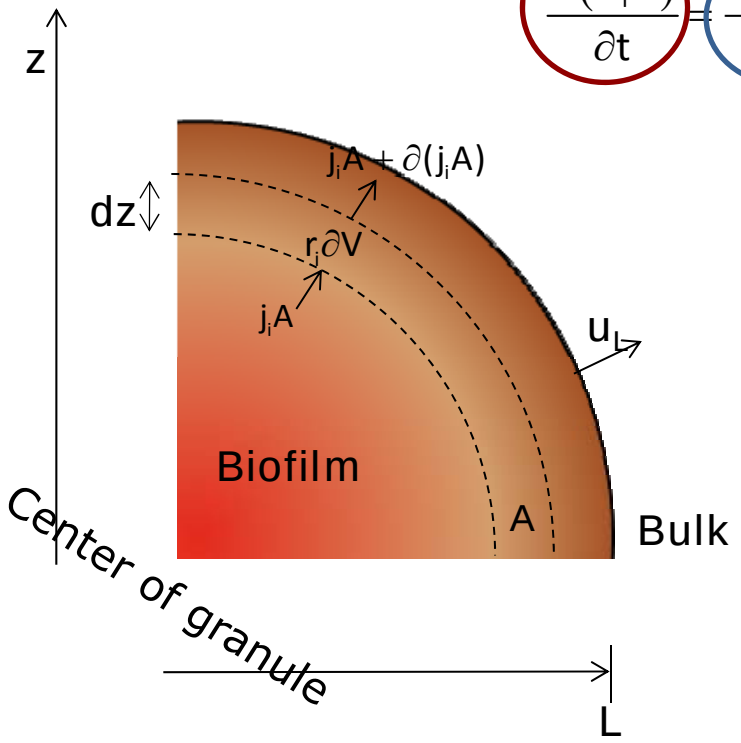


# Methods
# Model description

Biofilm mass balance equations – Transport and microbial metabolism

Accumulation = Inflow - Outflow + Generation - Consumption

$$\frac{\partial(C_i V)}{\partial t} = -\partial(j_i A) + r_i \partial V \quad <=> \quad \frac{\partial C_i}{\partial t} = \frac{1}{z^2}\frac{\partial}{\partial z}\left(z^2 j_{Ci}\right) + r_i$$




1. Transport of **soluble** compounds is governed by **diffusion** and of **particulate** compounds by **advection**:

$$j_{Si} = D_{bio,i}\frac{\partial S_i}{\partial x} \qquad j_{Xi} = -X_i u_F$$

2. The **granule radius** is a function of the growth and decay of bacteria and a detachment process:

$$\frac{dL}{dt} = u_{F,L} - u_D$$

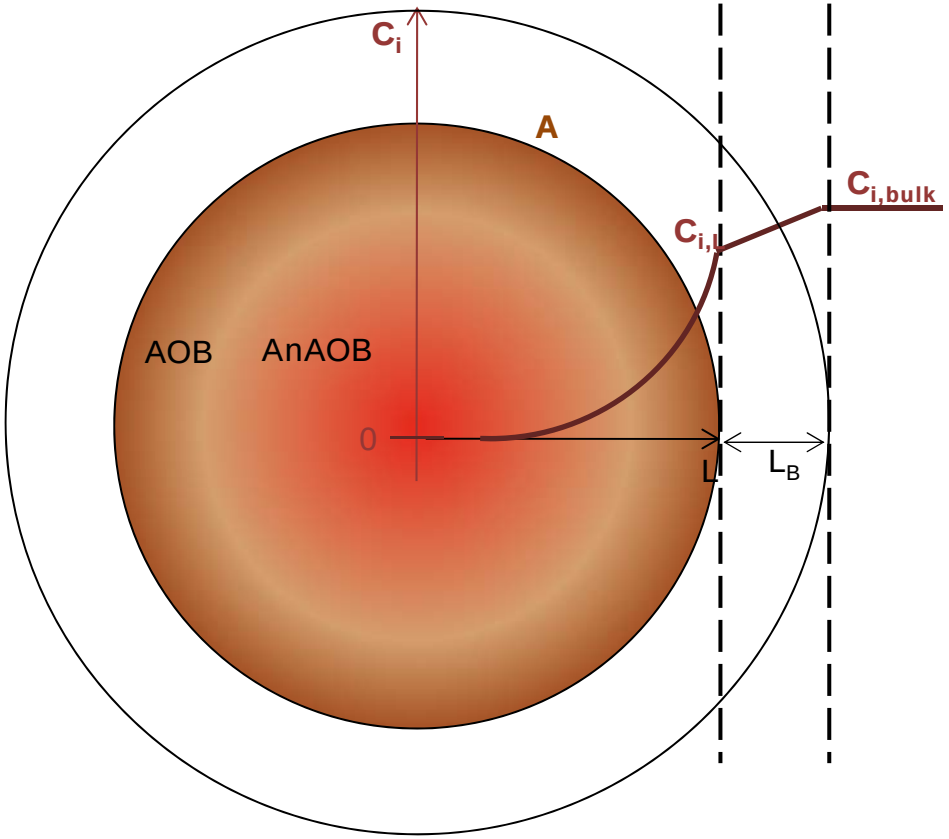
Where the advective velocity is a function of the growth of particulates on the "inside" of a given point k:

$$u_{F,k} = \frac{1}{A_k}\int_0^k A_k \left(\sum_{i=1}^{n_{part}} \frac{r_i}{\rho}\right)_k dz$$

# Methods
# Model description

Bulk liquid mass balance equations – Transport and microbial metabolism

Accumulation = Inflow - Outflow + Generation - Consumption




$$\frac{dC_i}{dt} = \frac{Q_{in}C_{i,in} - Q_{out}C_{i,bulk} - j_{Ci}A}{V} + r_{Ci}$$

Flux in and out of the biofilm:

Soluble species

$$j_{Si} = k_i\left(S_{i,bulk} - S_{i,L}\right)$$

Particulate species

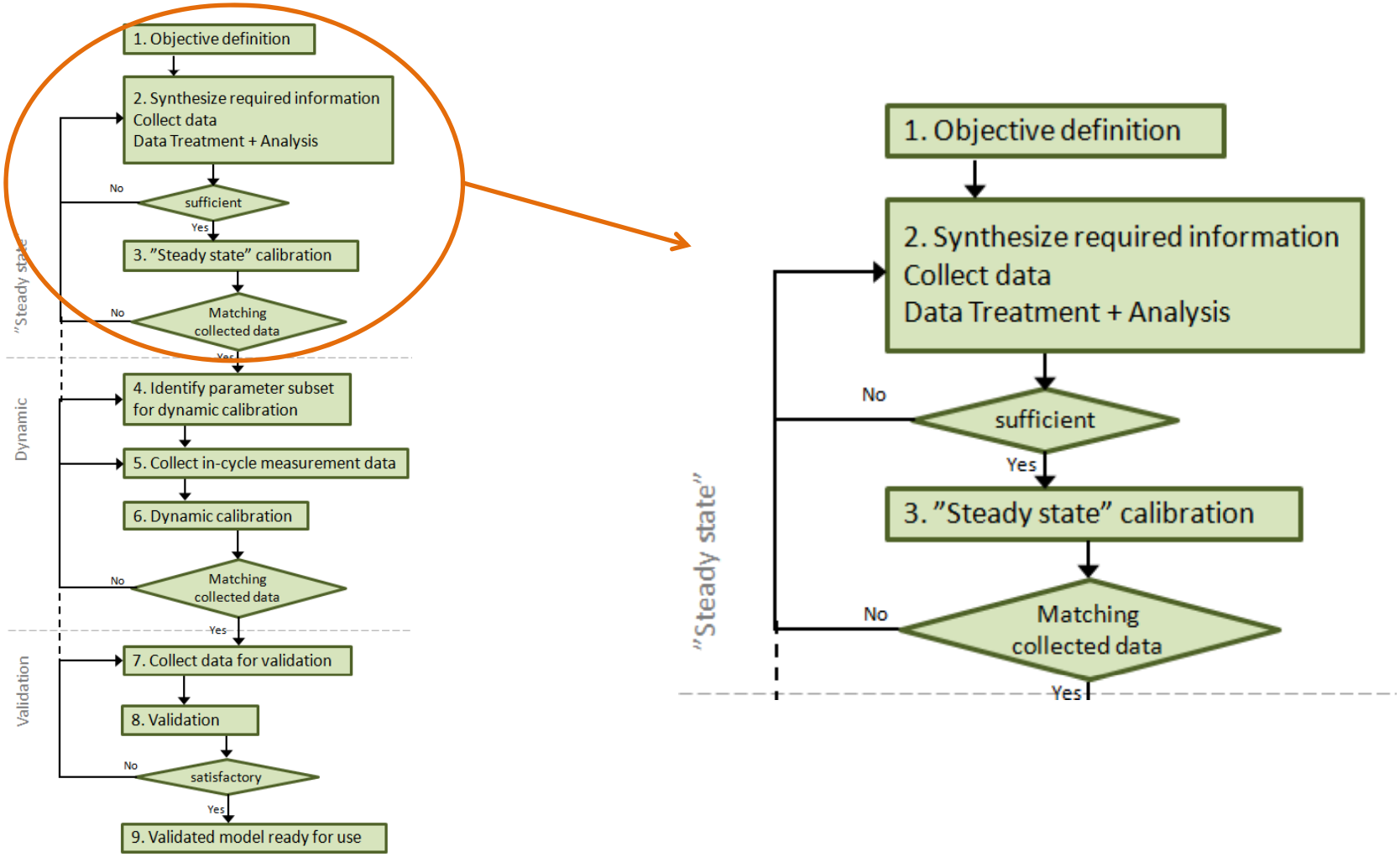
$$j_{Xi} = -u_D X_{i,L}$$

where

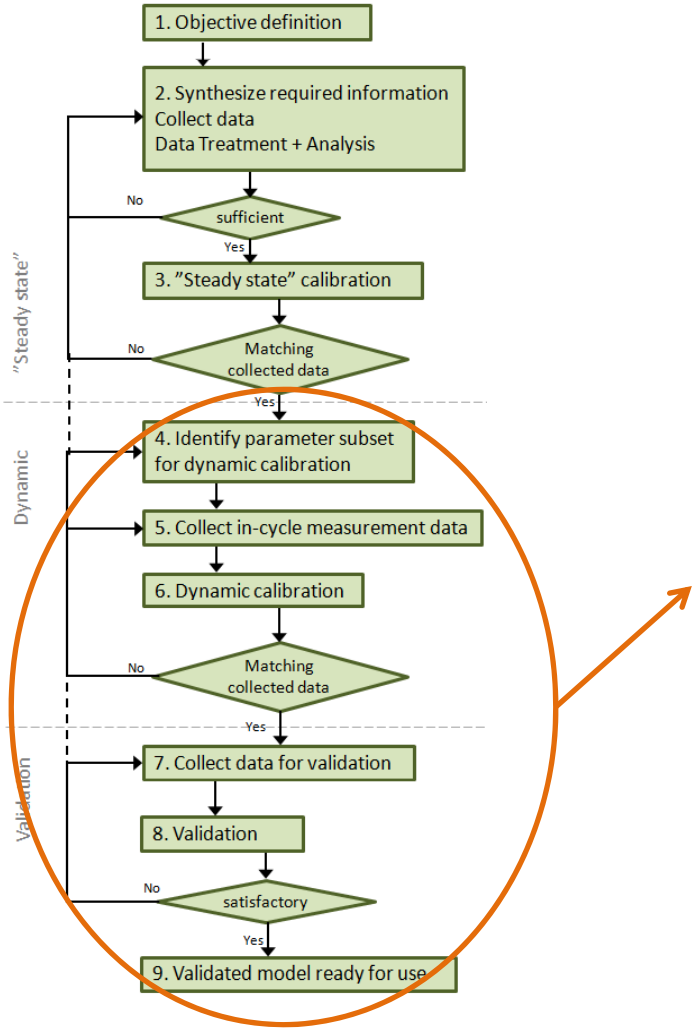
$$k_i = \frac{D_i}{L_B}$$

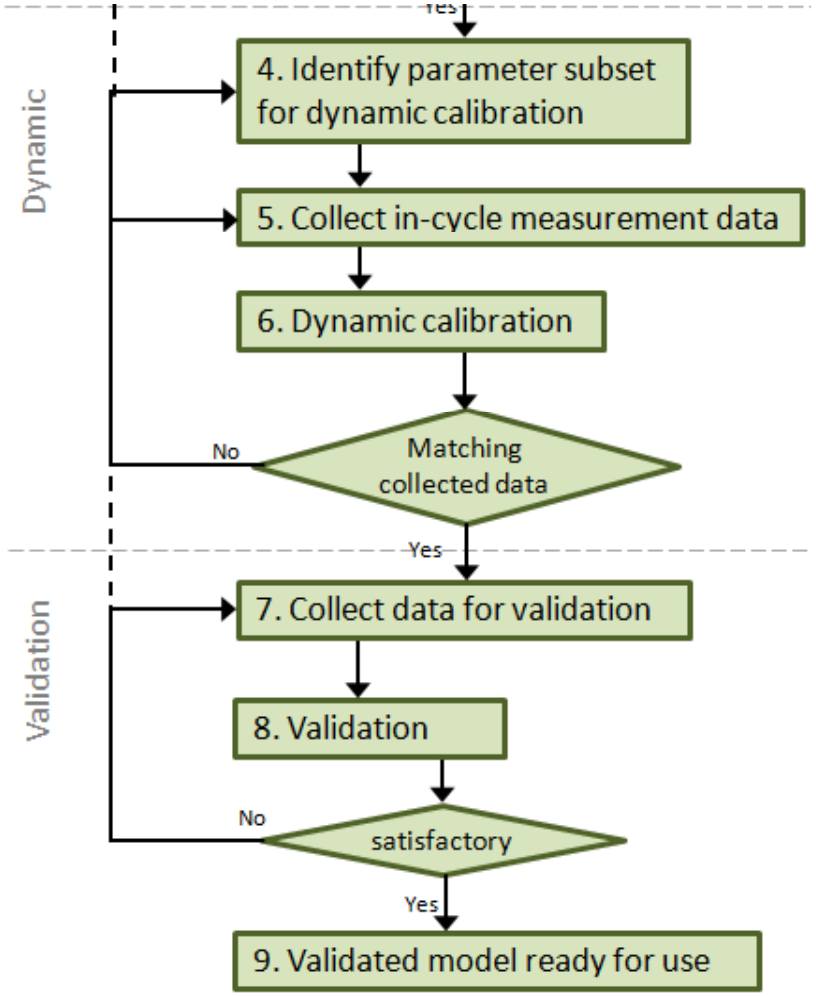
The mass transfer coefficient is estimated from a semi-empirical correlation considering mixing caused by bubble aeration

# Methods
# Methodology development



# Methods
# Methodology development



# Results
# Steady state calibration

- Step 1:

Determine bulk liquid soluble N species concentrations

- Step 2:

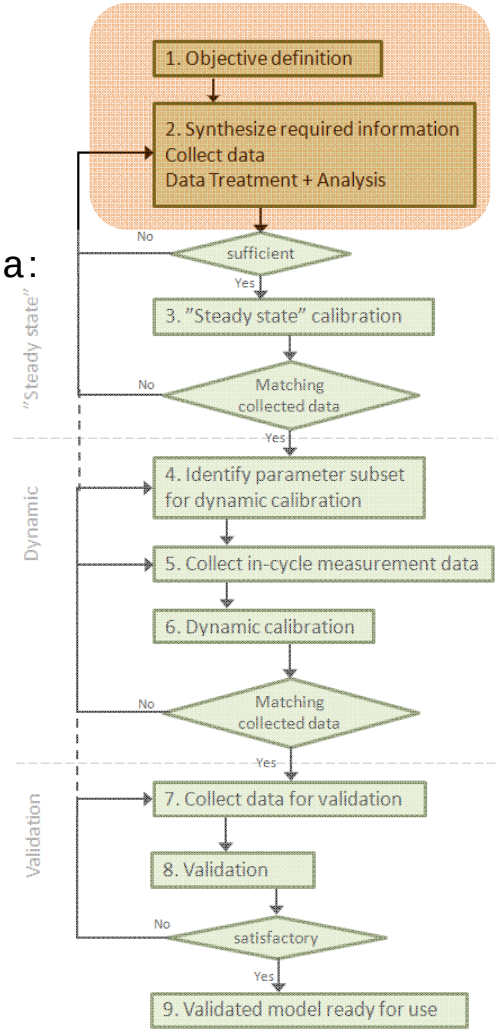
Capturing overall reactor performance through five evaluation criteria:
Three ratios and two efficiencies:

$$R1 = \frac{\Delta NO_2^-}{\Delta NH_4^+} \longrightarrow \text{AOB vs. AnAOB + NOB} \rightarrow \text{relative activity}$$

$$R2 = \frac{\Delta NH_4^+}{\Delta TN} \longrightarrow \text{AnAOB vs. AOB} \rightarrow \text{relative activity}$$

$$R3 = \frac{\Delta NO_3^-}{\Delta NH_4^+} \longrightarrow \text{AnAOB vs. NOB} \rightarrow \text{relative activity}$$

$$E1 = \frac{\Delta NH_4^+}{NH_{4,in}^+} \qquad E2 = \frac{\Delta TN}{TN_{in}} \longrightarrow \text{Absolute microbial activity}$$




*Mutlu et al., 2012. WWC Proceedings*

# Results
# Steady state calibration

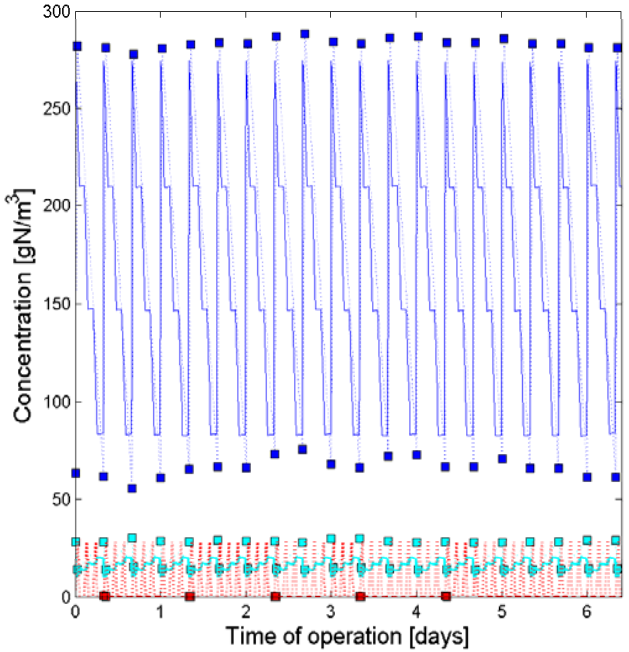
- Step 3:

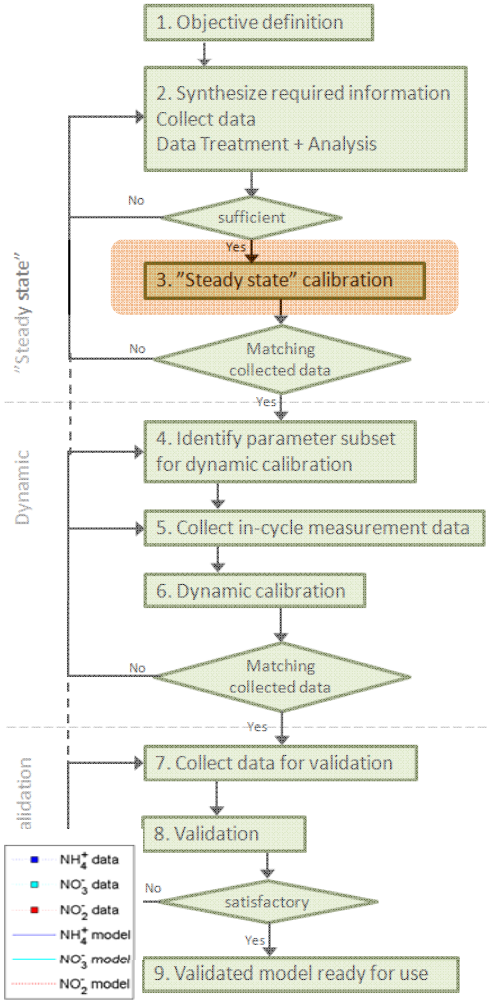
Since oxygen $k_La$ could not be experimentally estimated, this was calibrated based on the five evaluation criteria:

| | $k_La$ $d^{-1}$ | R1 $\Delta NO_2^-/\Delta NH_4^+$ | R2 $\Delta NH_4^+/\Delta TN$ | R3 $\Delta NO_3^-/\Delta TN$ | $NH_4^+$ removal % | TN removal % |
|---|---|---|---|---|---|---|
| Simulation | 524.4 | 0.000 | 1.052 | 0.049 | 79.25 | 74.32 |
| Experimental | - | 0.001 | 1.072 | 0.071 | 80.80 | 71.52 |

Experimental values were obtained as an average of one week of "steady state" operation.

Model was initialized by simulating continuous operation for 1000 days, which was then followed by 10 days of SBR operation of which the results from the last cycle were used for the steady state model evaluation.



# Results
# Dynamic calibration

- Step 4: Parameter subset identification

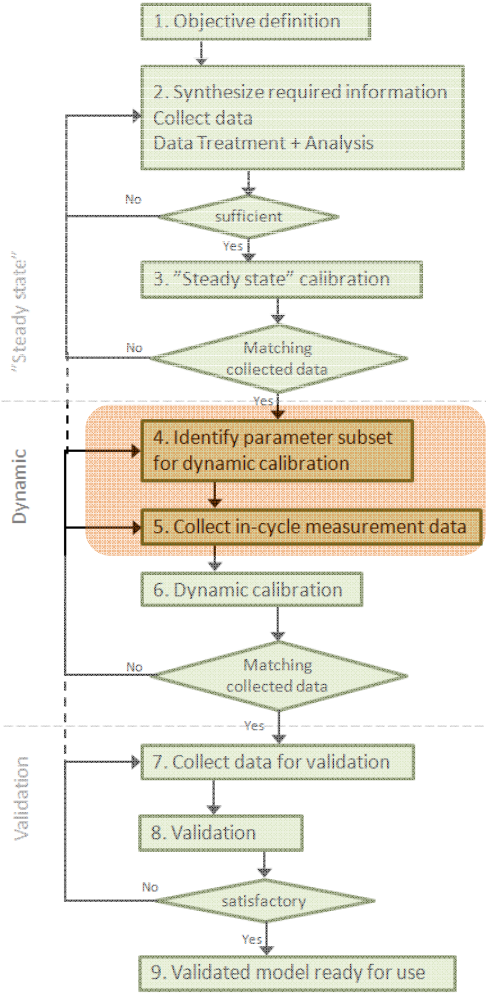
Based on global sensitivity analysis:

| | $\mu_{max,AOB}$ $d^{-1}$ | $K_{O2,AOB}$ $gCOD/m^3$ | $b_{AOB}$ $d^{-1}$ | $\mu_{max,AnAOB}$ $d^{-1}$ | $K_{O2,AnAOB}$ $gCOD/m^3$ | $Y_{AnAOB}$ gCOD/gN |
|---|---|---|---|---|---|---|
| Default value | 2.050 | 0.300 | 0.130 | 0.073 | 0.010 | 0.160 |
| Lower bound | 1.538 | 0.150 | 0.098 | 0.055 | 0.005 | 0.152 |
| Upper bound | 2.563 | 0.450 | 0.163 | 0.091 | 0.015 | 0.168 |

- Step 5: In-cycle data collection

Samples from bulk liquid were manually collected every 15 min. and analyzed for soluble N species.

Analysis results from three cycles were used for calibration.






*Vangsgaard et al., 2012. Bioresource Technol.*

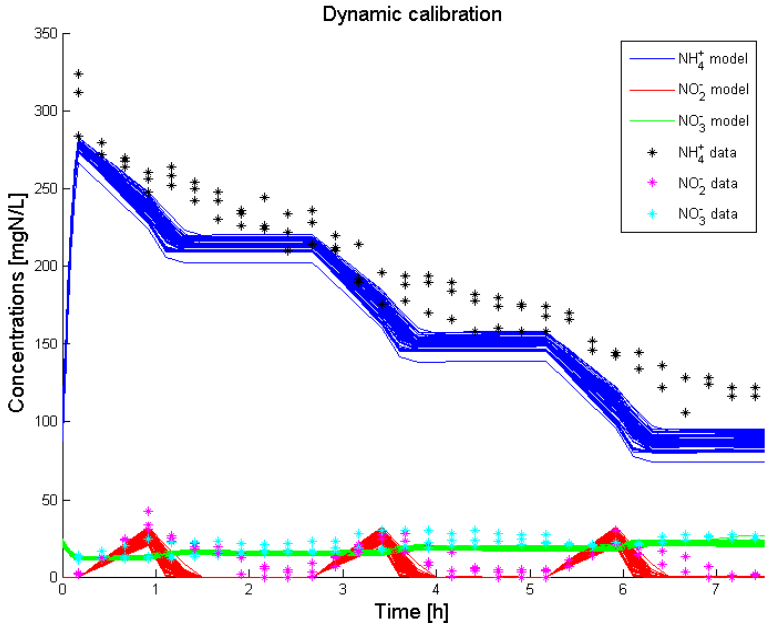
# Results
# Dynamic calibration

- Step 6: Calibration

Based on pragmatic Monte Carlo method, which was evaluated by WSSE:

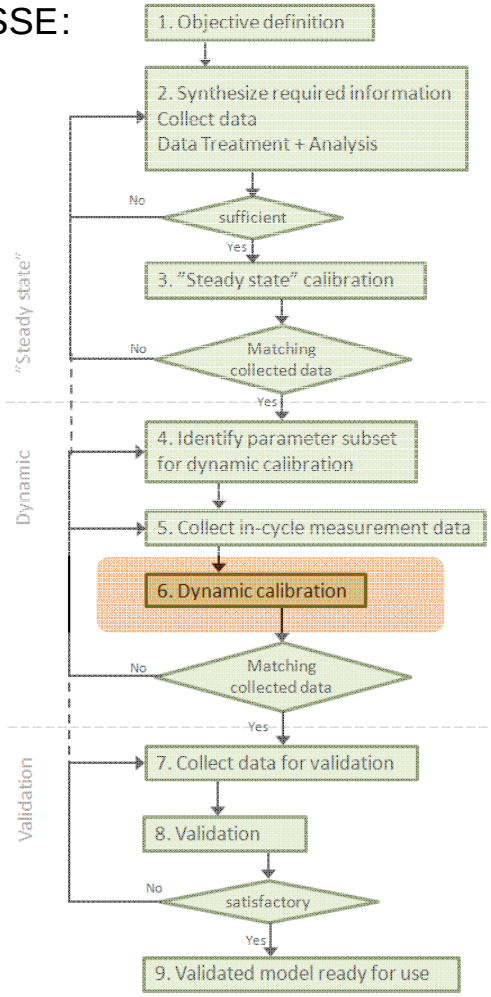
$$WSSE = \sum_{k=1}^{m}\sum_{i=1}^{n}\left(\frac{y_{meas,k}(t) - y_{model,k}(t,\theta)}{\sigma_k}\right)^2$$

| | $\mu_{max,AOB}$ $d^{-1}$ | $K_{O2,AOB}$ $gCOD/m^3$ | $b_{AOB}$ $d^{-1}$ | $\mu_{max,AnAOB}$ $d^{-1}$ | $K_{O2,AnAOB}$ $gCOD/m^3$ | $Y_{AnAOB}$ $gCOD/gN$ |
|---|---|---|---|---|---|---|
| Default value | 2.050 | 0.300 | 0.130 | 0.073 | 0.010 | 0.160 |
| Lower bound | 1.538 | 0.150 | 0.098 | 0.055 | 0.005 | 0.152 |
| Upper bound | 2.563 | 0.450 | 0.163 | 0.091 | 0.015 | 0.168 |
| Calibrated value | 2.450 | 0.165 | 0.136 | 0.068 | 0.011 | 0.166 |




However, all MC sims have an offset compared to the collected data

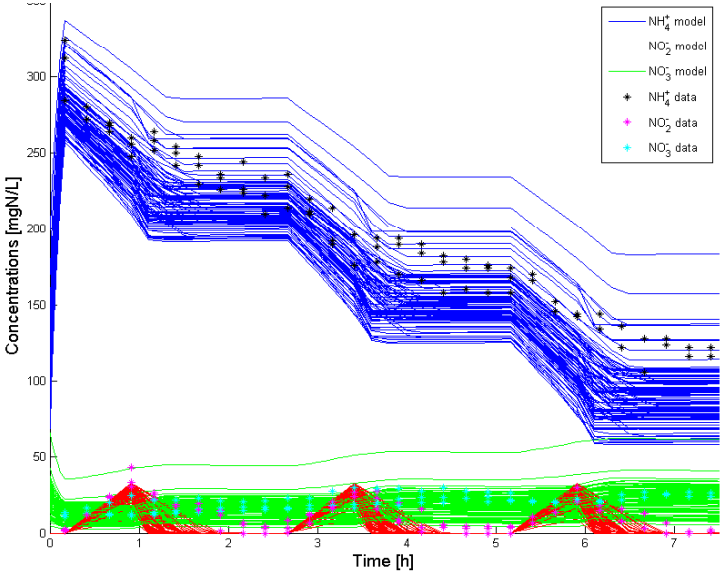
→ Iteration of step 4-6, in accordance with the methodology



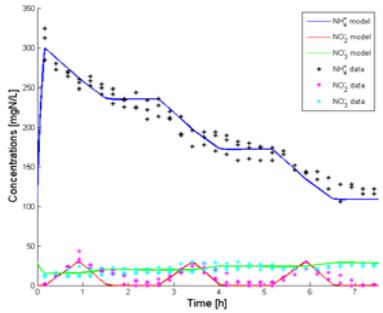
# Results
# Dynamic calibration

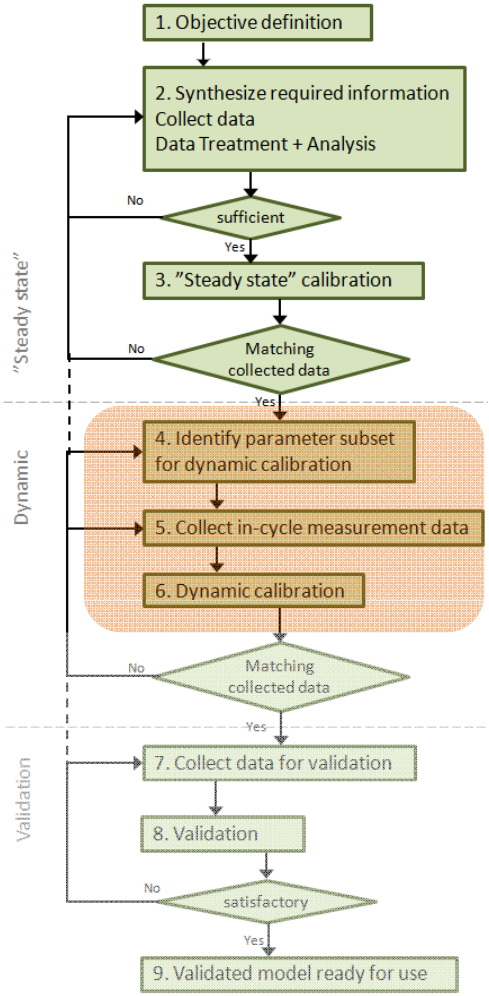
- Step 4-6 - iterated: Parameter subset and sampling space were expanded

| | Unit | Default value | Lower bound | Upper bound | Calibrated value |
|---|---|---|---|---|---|
| $\mu_{max,AOB}$ | $d^{-1}$ | 2.050 | 1.025 | 3.075 | 2.064 |
| $K_{O2,AOB}$ | $gCOD/m^3$ | 0.300 | 0.150 | 0.450 | 0.332 |
| $b_{AOB}$ | $d^{-1}$ | 0.130 | 0.065 | 0.195 | 0.150 |
| $\mu_{max,NOB}$ | $d^{-1}$ | 1.454 | 0.727 | 2.181 | 0.974 |
| $K_{O2,NOB}$ | $gCOD/m^3$ | 1.100 | 0.550 | 1.650 | 0.752 |
| $b_{NOB}$ | $d^{-1}$ | 0.061 | 0.030 | 0.091 | 0.069 |
| $\mu_{max,AnAOB}$ | $d^{-1}$ | 0.073 | 0.037 | 0.110 | 0.088 |
| $K_{O2,AnAOB}$ | $gCOD/m^3$ | 0.010 | 0.005 | 0.015 | 0.013 |
| $K_{HNO2,AnAOB}$ | $gN/m^3$ | 2.81e-6 | 1.41e-6 | 4.22e-6 | 2.92e-6 |
| $Y_{AOB}$ | gCOD/gN | 0.210 | 0.105 | 0.315 | 0.292 |
| $Y_{AnAOB}$ | gCOD/gN | 0.160 | 0.080 | 0.240 | 0.124 |
| $D_{NO2}$ | $m^2/d$ | 2.60e-4 | 1.30e-4 | 3.90e-4 | 1.70e-4 |
| $L_B$ | m | 1.76e-5 | 8.80e-6 | 2.64e-5 | 2.26e-5 |




Among the new MC sims, the subset sample giving the smallest error fitted much better to the data than the previous.



# Results
# Validation

- Step 7: Data collection for validation

Samples were collected during one cycle under slightly different conditions compared to the calibration cycles. The solids concentration was 4.4 g VSS/L and average granule size was 35 µm.

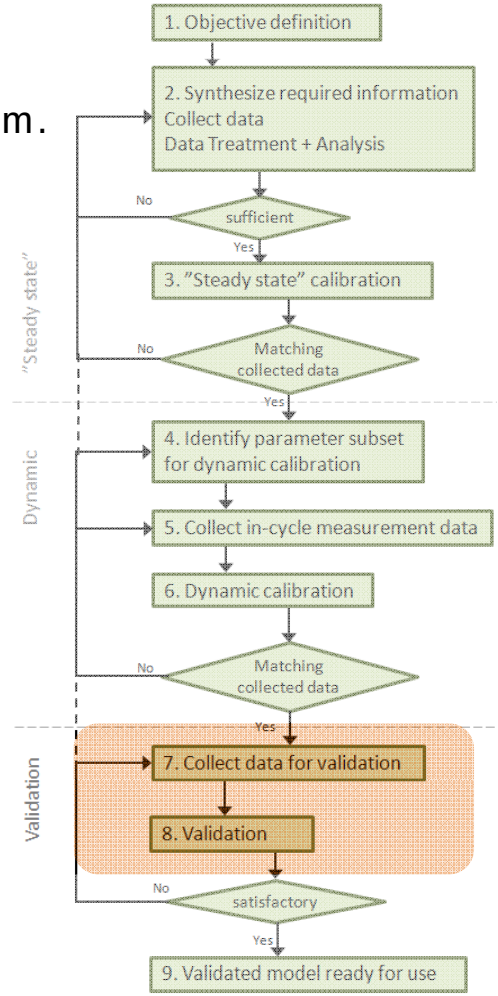
- Step 8: Validation

The validation was evaluated by the Janus coefficient:

$$J^2 = \frac{\frac{1}{n_{val}}\sum_{i=1}^{n_{val}}\left(y_{meas,i} - y_{model}(t_i,\theta)\right)^2}{\frac{1}{n_{cal}}\sum_{i=1}^{n_{cal}}\left(y_{meas,i} - y_{model}(t_i,\theta)\right)^2}$$

| | MAE | | RMSE | | Janus coefficient |
|---|---|---|---|---|---|
| Model output | Calibration | Validation | Calibration | Validation | |
| $NH_4^+$ | 0.030 | 0.053 | 0.039 | 0.057 | 1.478 |
| $NO_2^-$ | 0.265 | 0.116 | 0.366 | 0.173 | 0.473 |
| $NO_3^-$ | 0.131 | 0.080 | 0.171 | 0.093 | 0.544 |

J is relatively close to 1 for all model outputs, which implies a good model fit.



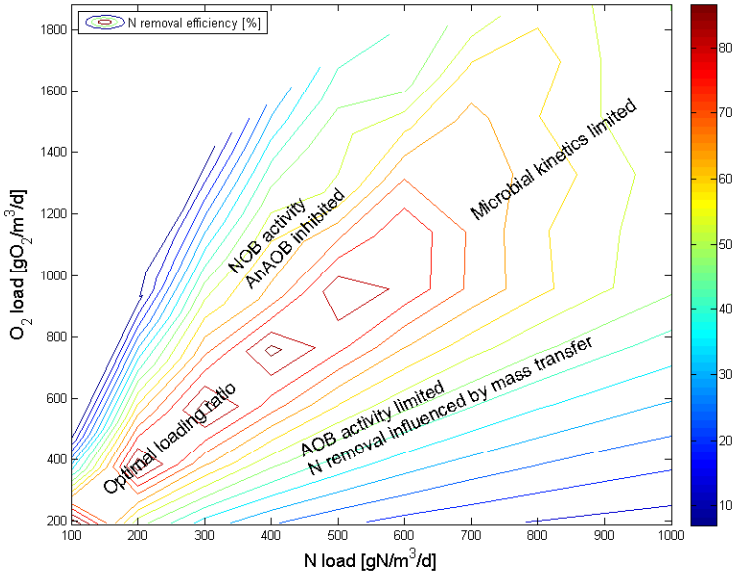
# Perspectives

- The validated model will be used for two purposes:

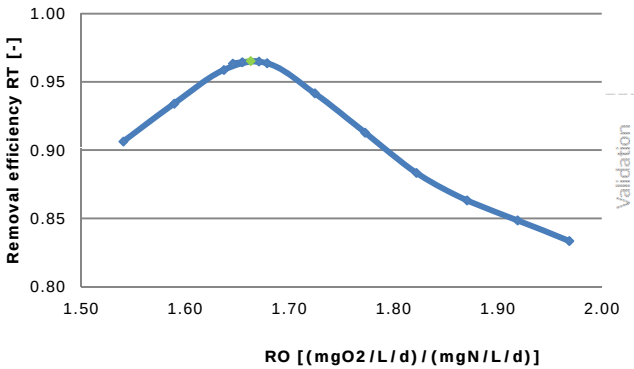
1) design of future lab-scale experiments in the form of perturbations in the operation

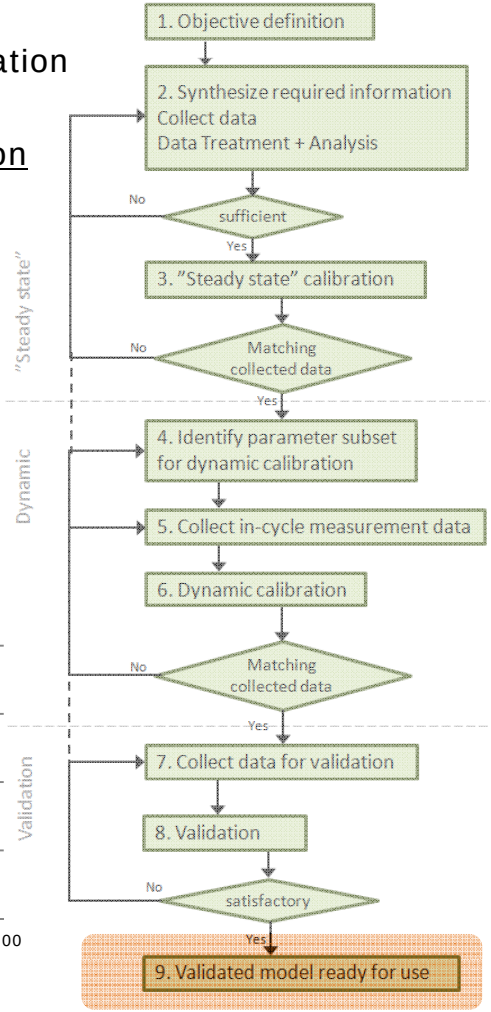
2) for predicting the process performance, which is important in future optimization and process control applications and in up-scaling of the system

Process optimization (based on results from Vangsgaard et al., 2012)




Analysis on calibrated model






*Vangsgaard et al., 2012. Bioresource Technol.*

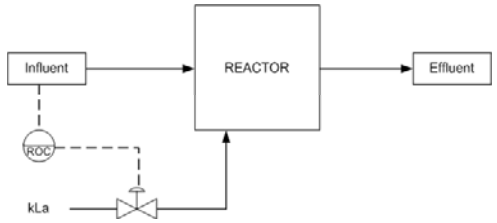
# Perspectives

2) for predicting the process performance, which is important in future optimization and process control applications and in up-scaling of the system

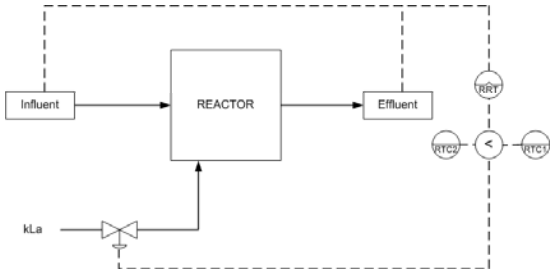
Control implementation:

Three different control strategies have been developed and analyzed on the calibrated model.

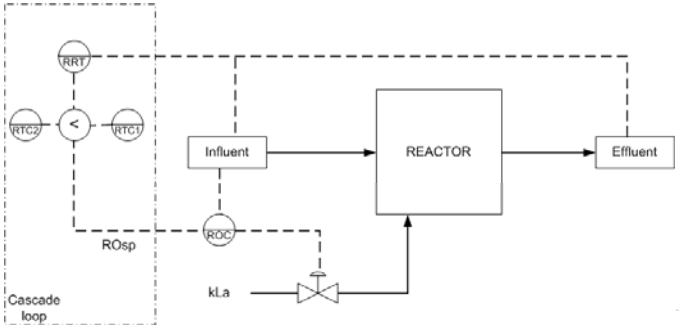
1. Feedforward based on oxygen/nitrogen loading ratio (Vangsgaard et al., 2012)



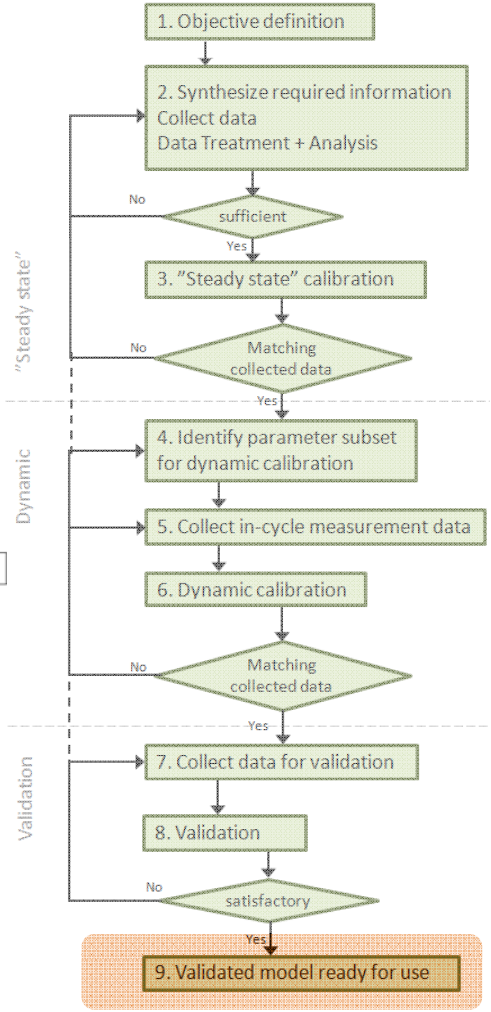

2. Feedforward + feedback based on stoichiometric rules (Mutlu et al., 2012):




3. Cascade control as a combination of 1. and 2.




As future extension of the current work, the developed control strategy will be experimentally tested in the lab-scale reactors.

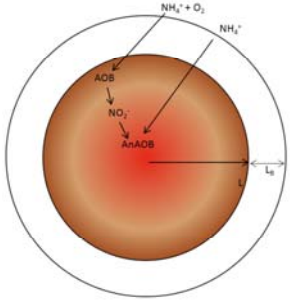


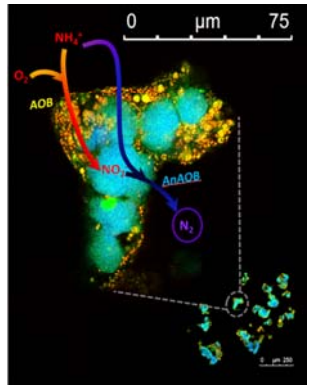



*Vangsgaard et al., 2012. Bioresource Technol.* *Mutlu et al., 2012. WWC Proceedings*

# Conclusions / wrap-up

- The model was successfully calibrated and validated by following a developed methodology:
  - First, $k_La$ was calibrated to long term "steady state" data by using novel evaluation criteria of **stoichiometric ratios** indicating the relative activity of the microbial groups.
  - Second, a subset of parameters were calibrated through dynamic calibration to in-cycle data.
  - An iteration of the second step was performed before a satisfactory result was obtained.
- A fast and efficient novel **initialization process** was developed
  - Simulating 1000 days of continuous operation before SBR operation.
- The model is now being used for optimization and control structure analyses.



# Thanks for your attention!

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