

# Coating Fine Particles

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# Aim of this work

- Is the coating process scalable?
- Low leakage of the active during the coating process
- Optimization
- Documentation



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# Background

New film    Aged film



Paint without biocide

Paint with biocide – state-of-the-art

Paint with encapsulated biocide



# What parameters have an effect on the release rate?

- Diffusion of the active out of the pores
- Shell thickness
- Solubility of the active in the coating
- Porosity of the coating
- The size of the particle → different diffusion lengths

# Materials

Material	Supplier	Properties of interest
Porous silica SD 4859	PQ Corporation	Particle size 3 $\mu\text{m}$ , Pore volume 2 mL/g
Sodium dodecyl sulfate (SDS)	Sigma Aldrich	288 g/mole
Polyethylene imine (PEI)	Aldrich chemistry	1800 g/mole
Tetraethyl orthosilicate (TEOS)	Aldrich chemistry	98 % 208 g/mole Negatively charged above pH 2-3
Ortho-vanillin	Aldrich chemistry	152 g/mole, Solubility 4.6 g/L water
Hydrochloric acid	VWR, Analar Normapur	37 %
Ethanol	Solveco	95 %

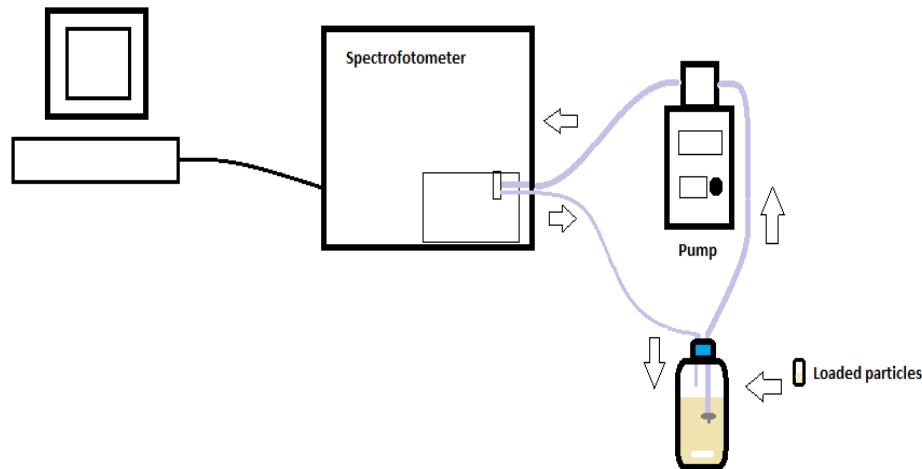
# Methods

1. Loading of particles
2. PEI coating
3. TEOS coating
4. Increasing particle concentration



# Analysis

- TGA - adsorption isotherm
- UV-VIS - controlled release (sink conditions)



# Experiments - overview

- Loading of particles
- PEI coating on loaded and empty particles
- The active dissolved in coating solutions
- TEOS coatings on loaded, PEI coated composites
- Increased particle concentration in PEI dispersions





# Loading of particles

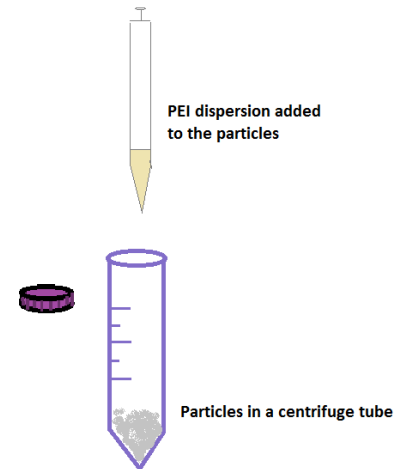
## Loading of particles

- Vanillin dissolved in acetone
  - Particles added
  - Stirring and ultrasonication
  - Filtration
- 
- TGA results showed that about 35 % of the composite is vanillin

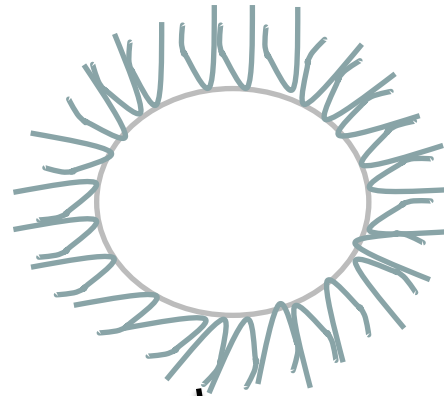
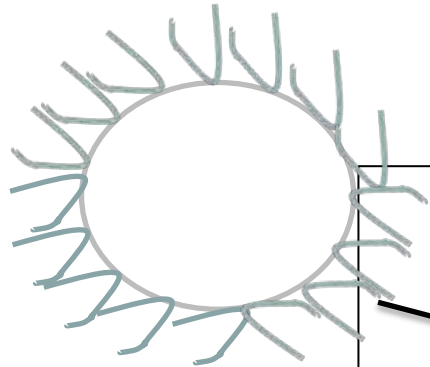
# PEI – coating process

## PEI coating of particles

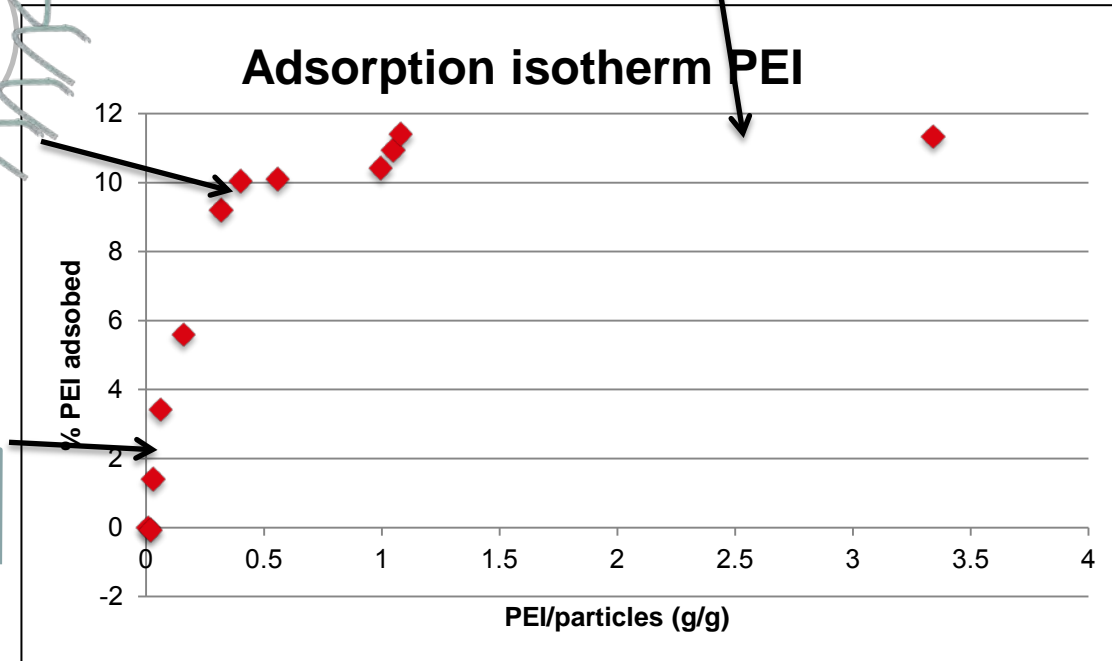
- Disperse PEI in water (0.1 M NaCl) with or without vanillin present
- Add PEI dispersion to particle containing centrifuge tubes (5 % particle concentration) and shake tubes
- Leave tubes at shaking table over night
- Centrifuge, wash and dry



# PEI – empty particles

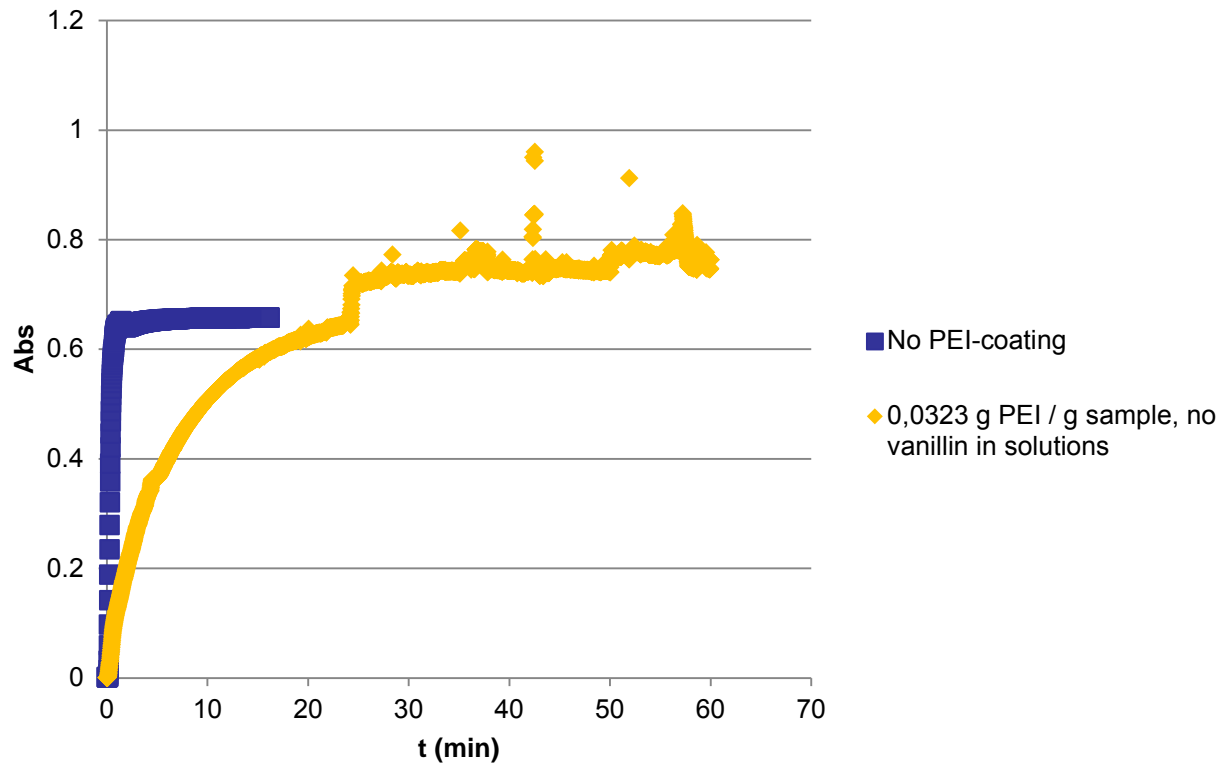


Adsorption isotherm PEI



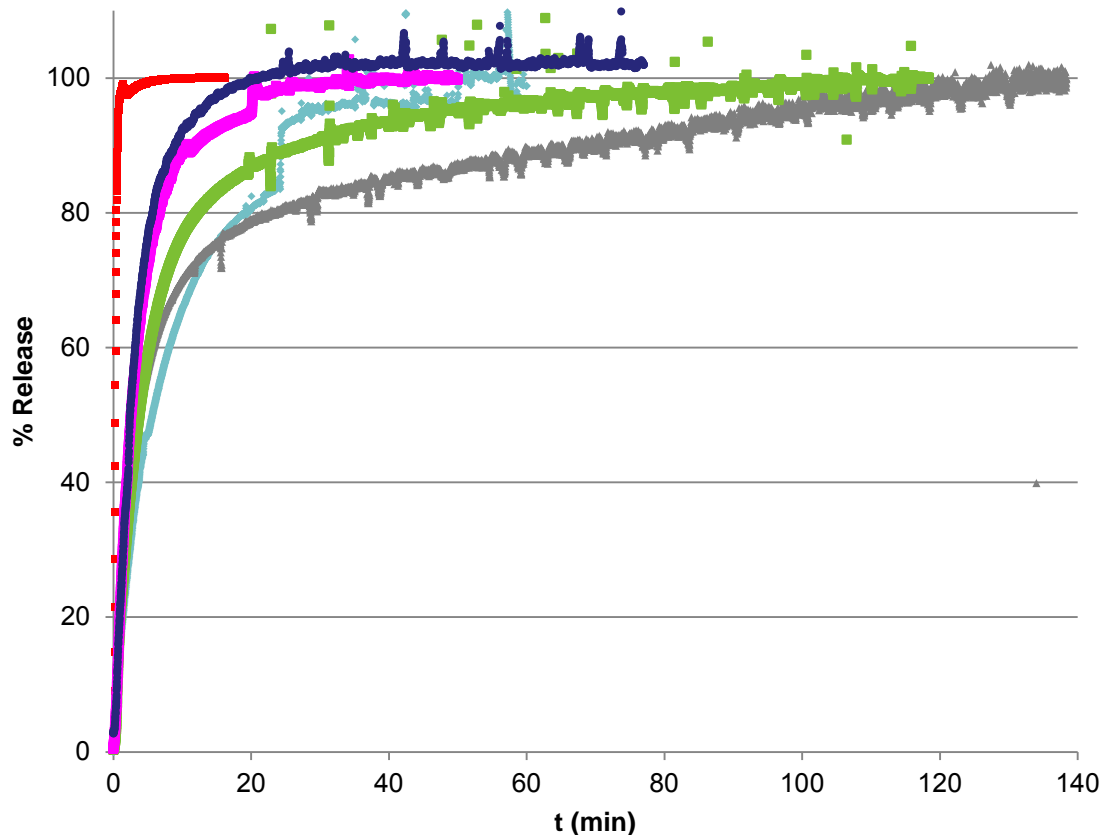
# PEI – loaded particles

## Spectroscopy, 262 nm



# PEI – loaded particles

## Release – no vanillin in loading solution



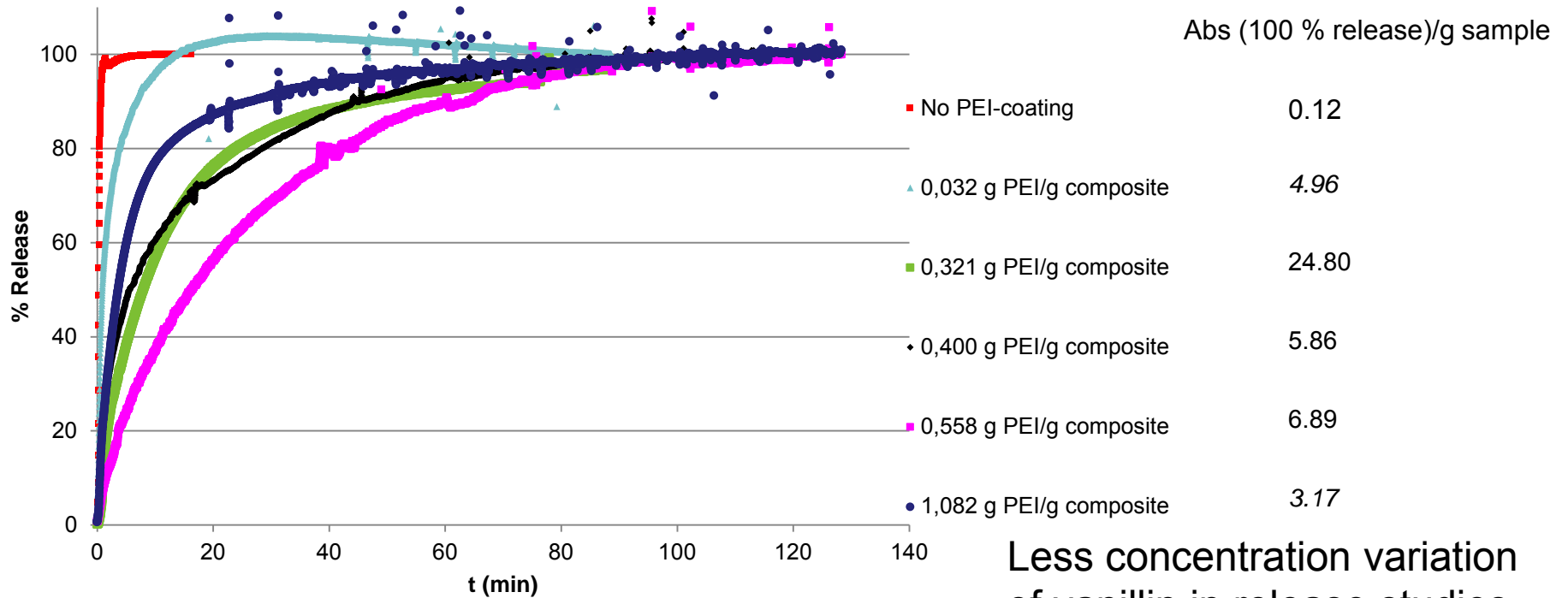
Abs (100 % release)/g sample

■ No PEI-coating	0.12
◆ 0,03 g PEI/g composite	8.07
▲ 0,159 g PEI/g composite	12.42
■ 0,3196 g PEI/g composite	3.11
■ 0,5653 g PEI/g composite	0.16
● 1,054 g PEI/g composite	5.40

Large concentration variation  
of vanillin in release studies

# PEI – loaded particles

## Release - vanillin in coating solutions



Less concentration variation  
of vanillin in release studies

# PEI - summary

- PEI adsorbs on the particle surface
- Complete release without coating 1-2 minutes
- Complete release with PEI coating 20-60 minutes
- Maximum adsorption of PEI is about 10 % of composite particle weight
- Different types of adsorption with different PEI/particle ratios
- Wide distribution of absorbance data as the active is not dissolved in coating solutions
- Narrower distribution of absorbance data as the active is dissolved in coating solutions
- More repetitions necessary

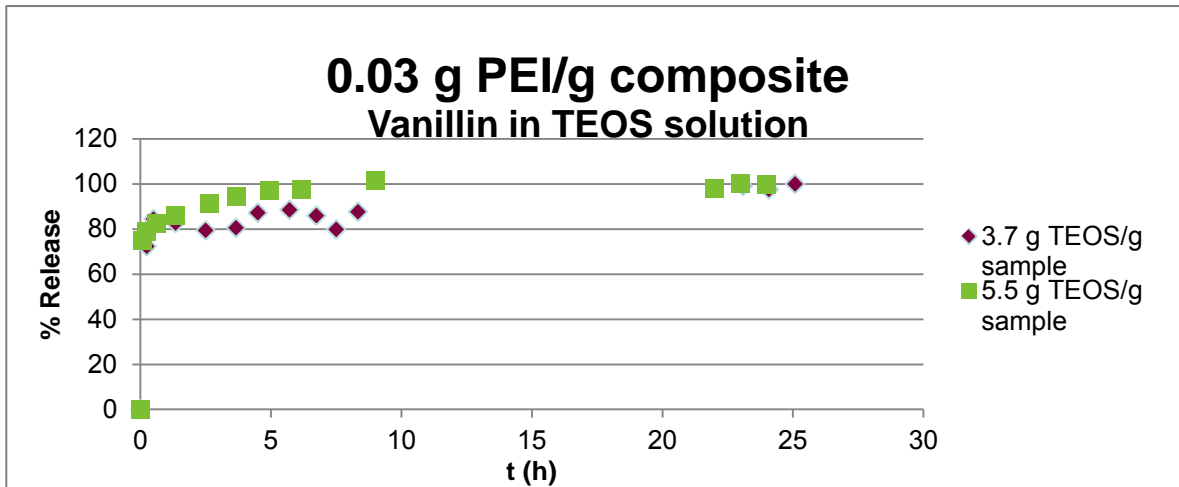


# TEOS – coating process

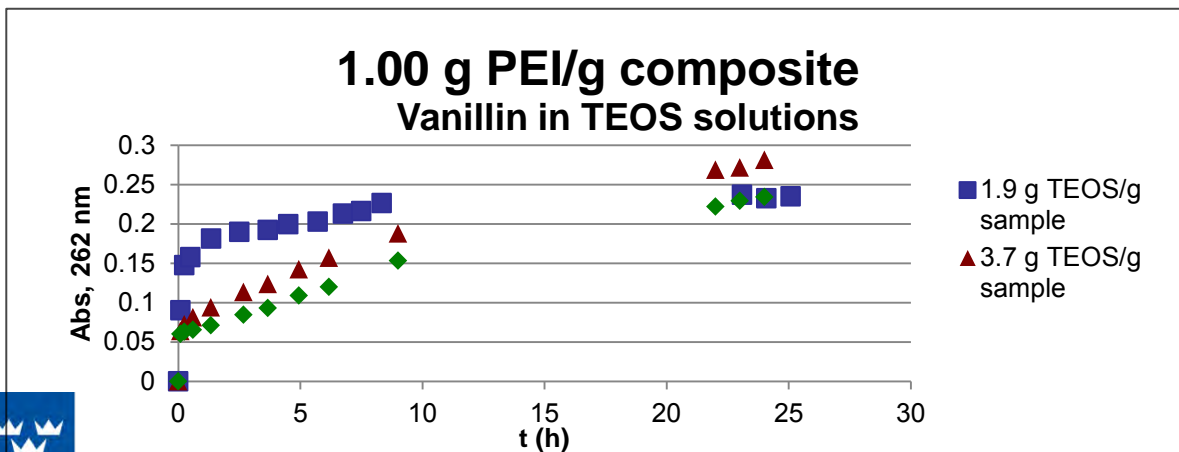
- TEOS solution on PEI coated composites
- Shake until homogenous solution
- Adjust pH
- Centrifuge, wash and dry



# TEOS – release rate



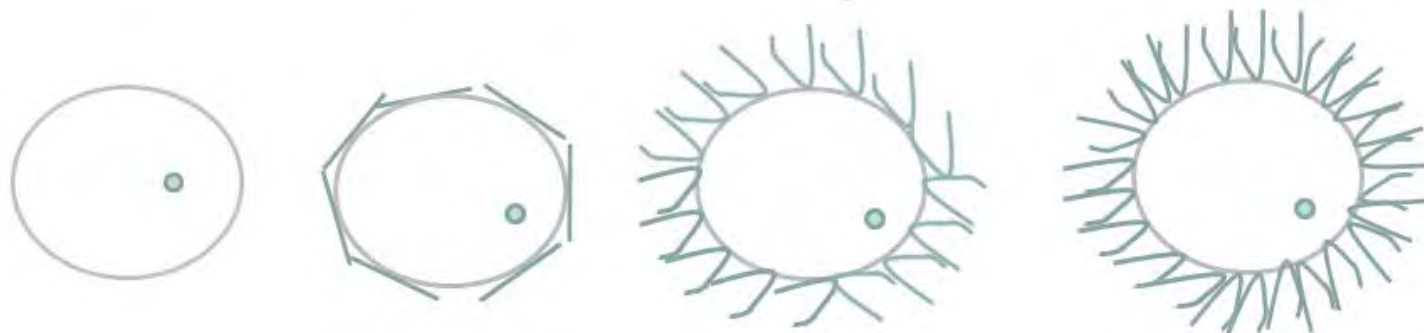
Vanillin released within 1 h



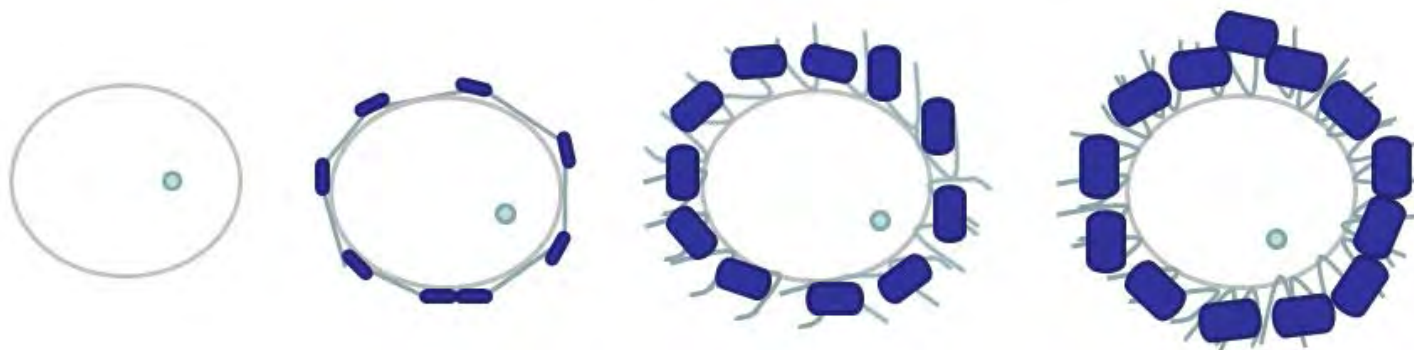
Vanillin not completely released within 24 h

# TEOS

PEI coating



PEI and  
TEOS coating



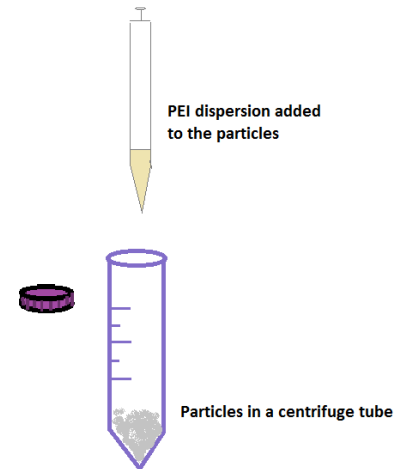
# TEOS – summary

- Lower release rate with TEOS on PEI coated particles
- Release of the active even after 24 hours
- No encapsulation at lowest amount PEI and TEOS on the composites



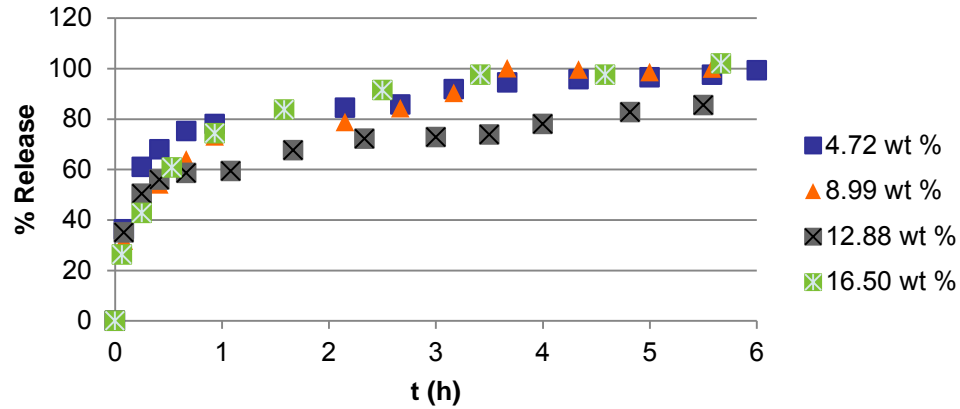
# Increased particle concentration

- Original concentration 4.7 %
- Increased concentration to 9.0 %, 12.9 % and 16.5 %
- No TEOS in this test only loaded Particles and PEI

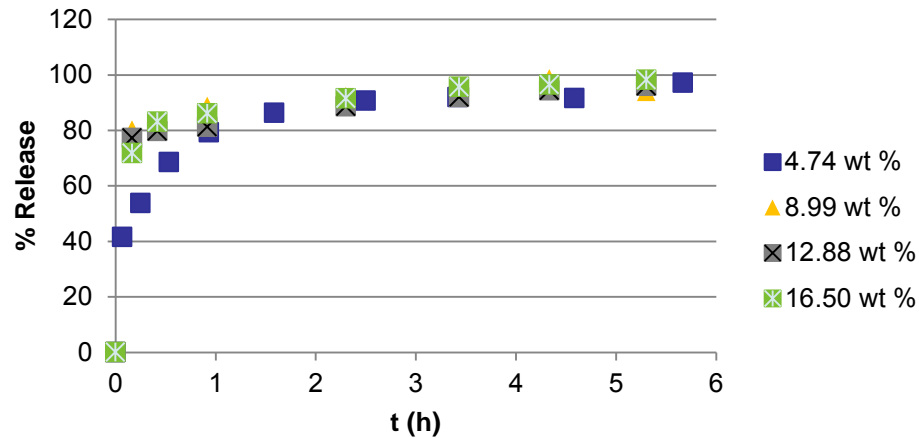


# Increased particle concentration

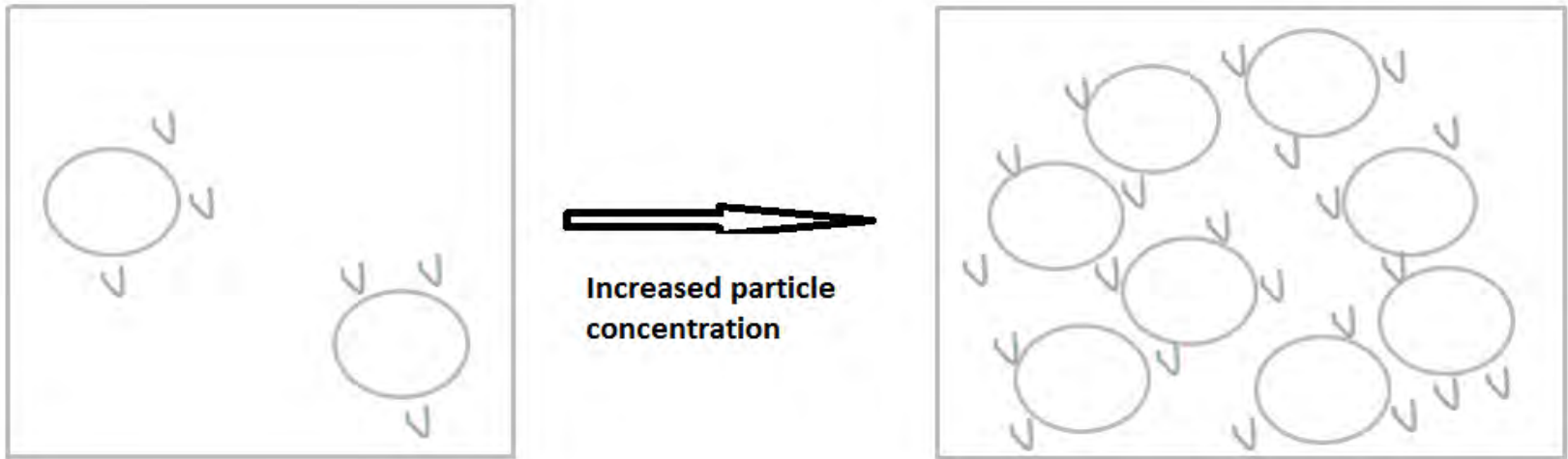
## 0,6 PEI/composite (g/g)



## 1,1 PEI/composite (g/g)



# Increased particle concentration



The likelihood for agglomeration increases as concentration increases

- Heteroflocculation
- Patch flocculation

# Increased particle concentration - summary

- It is possible to increase the particle concentration
- Reproducible release measurements for both PEI/composite ratios and all concentrations

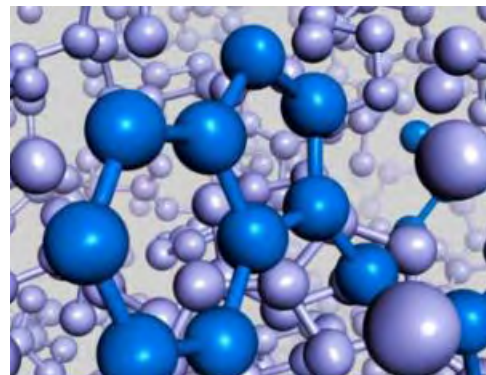


# Conclusions

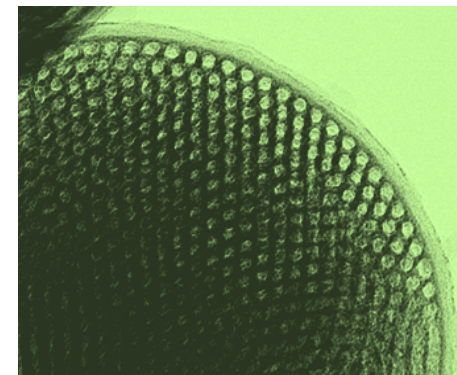
- PEI adsorbs on the particle surface. Maximum about 10 % of (empty) composite particle weight
- Vanillin in coating solution reduce release of vanillin from composite particles
- For a release time at about 20-60 min → Use PEI coatings
- For a release time at more than 24 h → Use PEI and TEOS coatings
- Increased particle concentration feasible







Protein and peptide formulation



Controlled delivery and release

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**We assist in need-driven research and innovation in the formulation area.**

The mission of the work within the platform is to improve performance of formulations for applications in a wide range of industrial areas resulting in **sustainable products, reduced side-effects and improved health.**

### CONTACT:

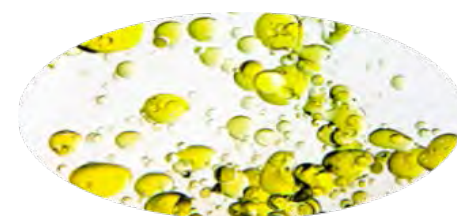
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Powder technology



Emulsions and dispersions