Extraction Column design for high viscosity systems

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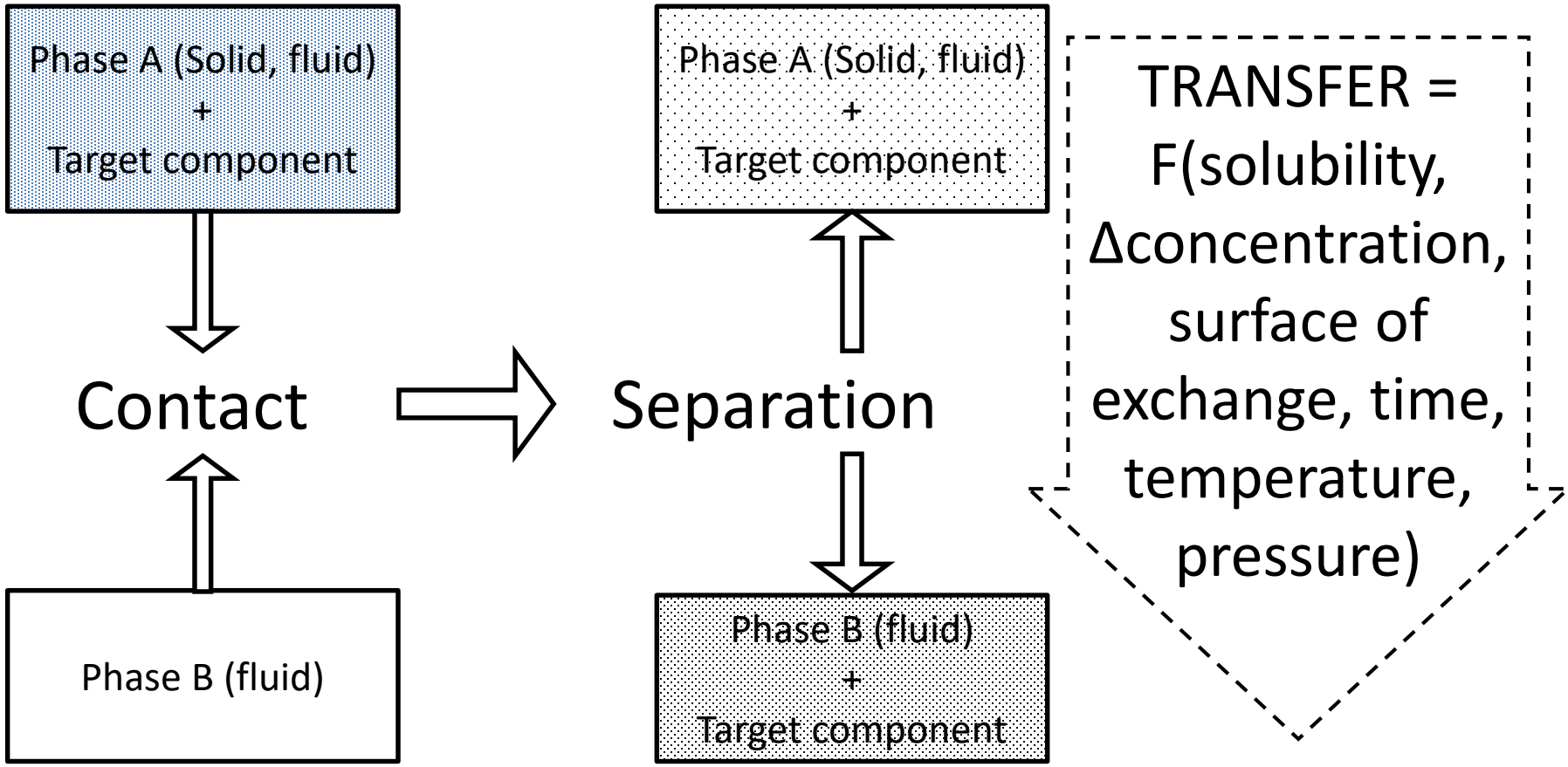
Presentation Plan

1. Context and motivation
2. Goal of the project
3. Experimental aspect
4. Programming aspect

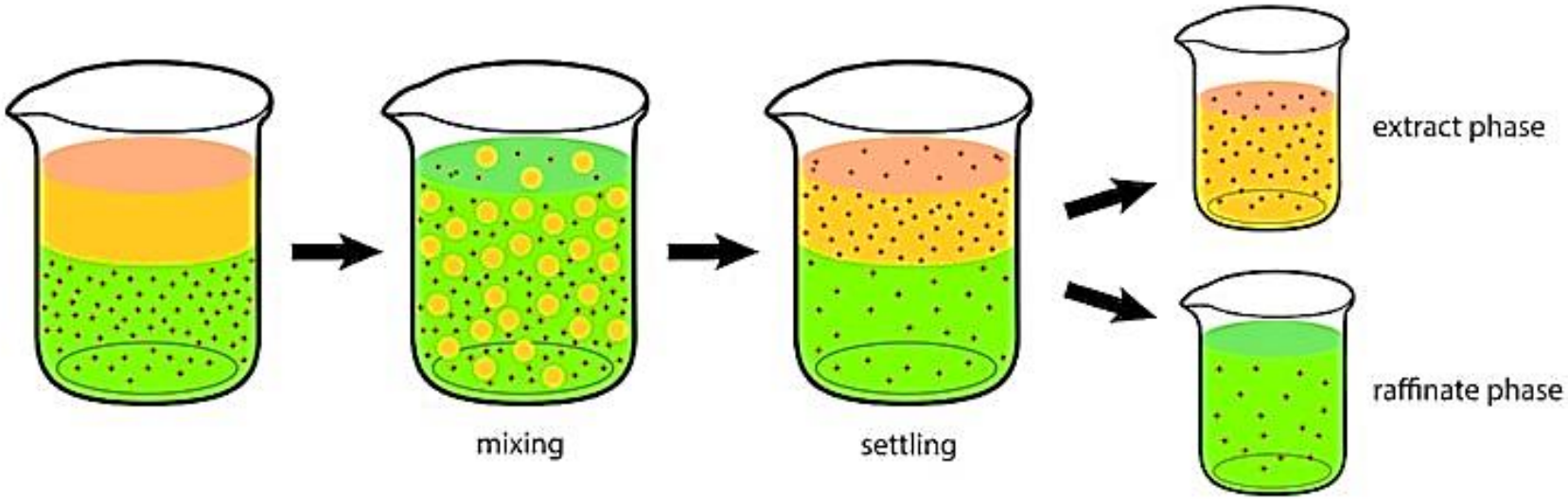
1. Context and Motivation

Extraction: separation process



1.2. Phenomena influencing solvent extraction and their interactions

Liquid - Liquid Extraction



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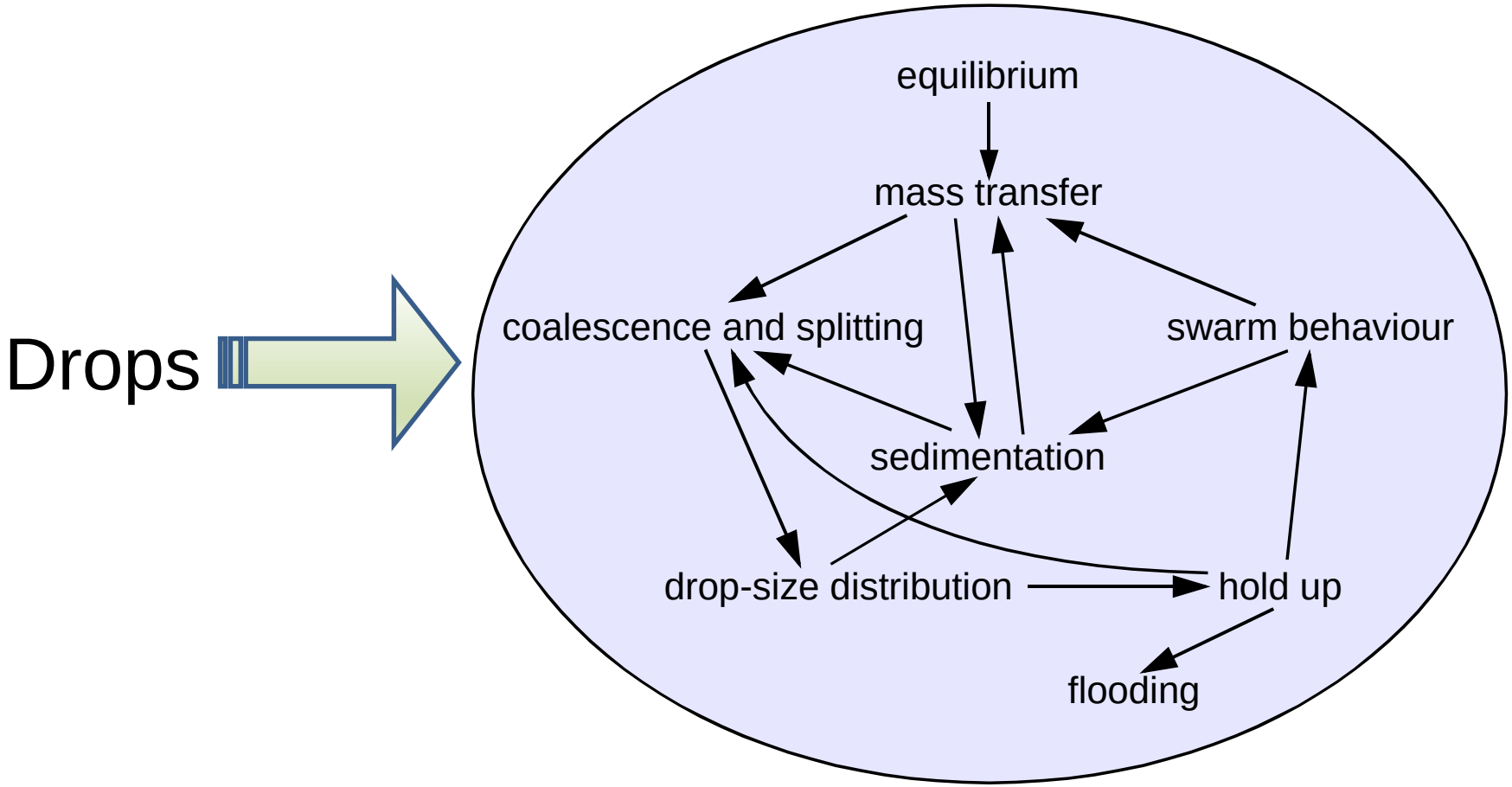
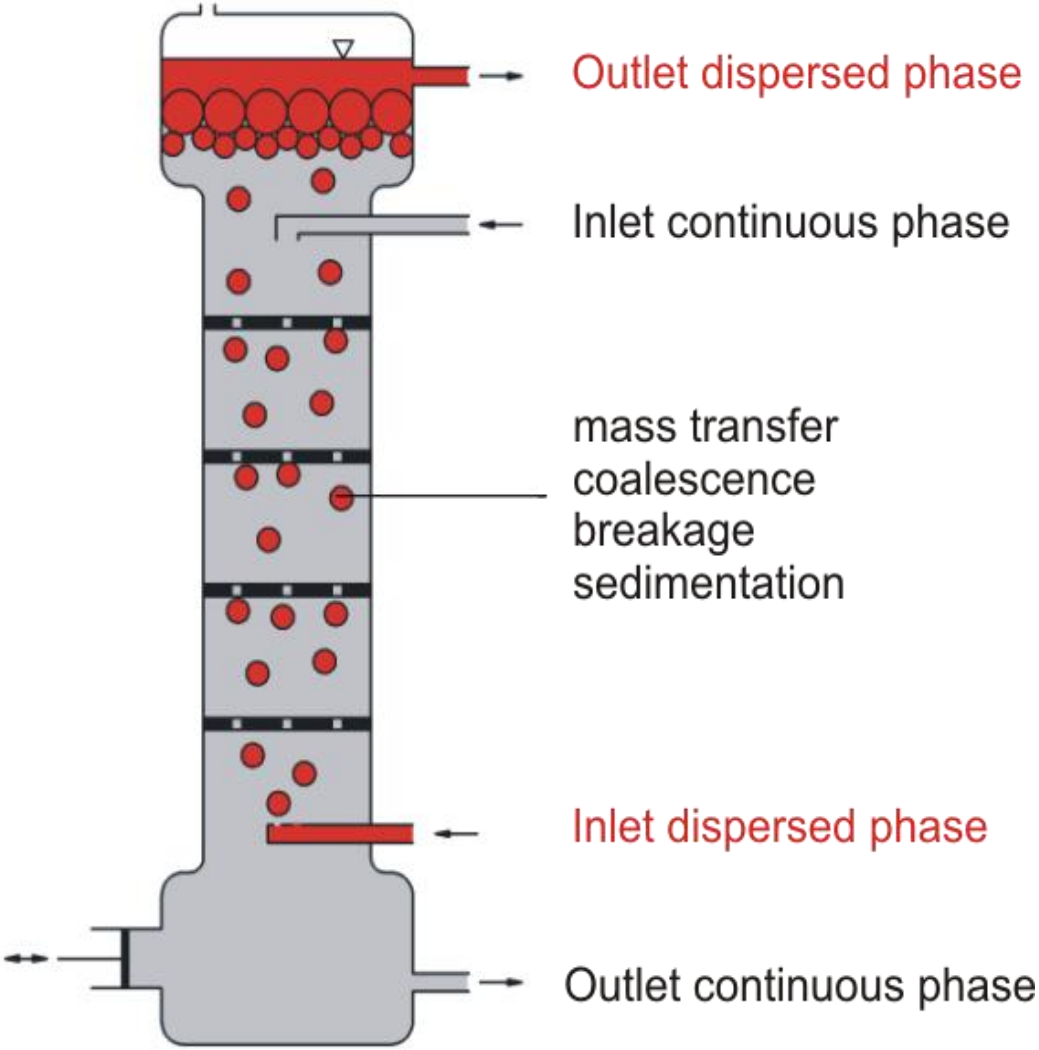


1.3. Extraction columns design

Liquid - Liquid extraction column design



1.3.1. Nature of the materials

- Change from fossil based to bio-based raw material

- Increase in oxygen content and stronger molecular interactions;
- Lower vapor pressures of the mixtures;
- Higher viscosity.

1.3.2. Extraction columns design

- Based on pilot-plant experiments

- Requires experience and big amount of chemicals;

- Time consuming and expensive.

1.3.3. Extraction columns

- Design of extraction columns based on lab-scale experiments: **single drop experiments**;

- Development and scale-up of extraction processes.

1.4. ReDrop (REpresentive DROPs)

- Simulation (simulation tool/software): mathematical models;

☞ **<u>ReDrop</u>**: Based on single drops experiments and study.

✓ Describe and predict all drop phenomena with great precision.

1.4. ReDrop (REpresentive DROPs)

❗ Models validated for viscosities of around 1 mPas and 100 mPas.

☞ Investigation need to complete drop behaviour description from 1 to 100 mPas.

2. Goal of the project

Optimization of extraction columns design, with a focus on increased viscosity systems.

2. Intermediate goals

- Validation of suitable models using single drop experiments (Lab-scale) from 1 to 100 mPas for both phases: Sedimentation and mass transfer;

- Rewrite ReDrop and add the obtained models to increase its usability;

- Validation of ReDrop simulations at Pilot/Industrial scale.

3. Experimental aspect

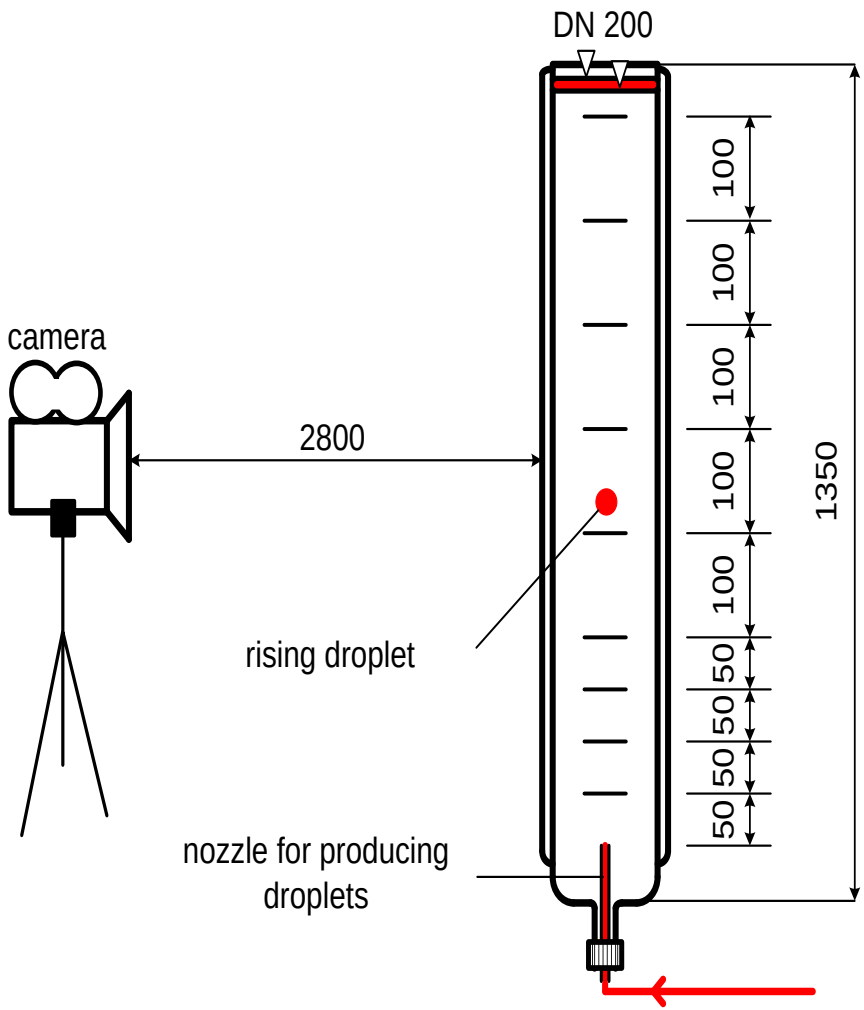
- Material: Modified Standard liquid-liquid extraction system (EFCE) using polymers to increase the phases viscosities;

  - Continuous phase: Water + PEG;
  - Transfer component: Acetone;
  - Dispersed phase: Toluene + Paraffin oil.

- Phenomena to be studied:

  - Sedimentation;
  - Mass transfer.

3.1. Sedimentation experiments



- Lab scale single drops experiments at different viscosities both phases ;
- Model validation;
- Integration of the results in ReDROP

3.1.1. Some of the models to be studied

Models	Equations
Stockes	$\mathbf{V}_\infty = \left(\frac{\mathbf{4dg\Delta\rho}}{\mathbf{3\rho_c C_w}}\right)^{\frac{1}{2}}$
Klee and Treybal (1956)	$\mathrm{d_{crit}} = 0.3\rho_c{}^{0.14}\Delta\rho^{-0.43}\eta_c{}^{0.3}\sigma^{0.24}$ $\mathbf{V}_\infty = \mathbf{38.3\rho_c}^{-0.45}\Delta\boldsymbol{\rho}^{0.58}\boldsymbol{\eta_c}^{-0.11}\boldsymbol{d}^{0.7}\ (\mathbf{d < dcrit})$ $\mathbf{V}_\infty = \mathbf{17.6\rho_c}^{-0.55}\Delta\boldsymbol{\rho}^{0.28}\boldsymbol{\eta_c}^{0.1}\boldsymbol{\sigma}^{0.18}\ (\mathbf{d > dcrit})$
Vignes (1965)	$\mathbf{V}_\infty = \frac{\boldsymbol{d}}{\mathbf{4.2}}\left(\frac{\mathbf{g\Delta\rho}}{\boldsymbol{\rho_c}}\right)^{\frac{2}{3}}\left(\frac{\boldsymbol{\rho_c}}{\boldsymbol{\eta_c}}\right)^{\frac{1}{3}}\left(\mathbf{1} - \frac{\mathbf{E\ddot{o}}}{\mathbf{6}}\right)$
Misek (1974)	$\mathbf{V}_\infty = \mathbf{0.249d}\left(\frac{\mathbf{g^2\Delta\rho^2}}{\mathbf{\rho_c\eta_c}}\right)^{\frac{1}{3}}$
Grace et al. (1976)	$C_{Grace,2} = \frac{4}{3}E\ddot{o}Mo^{-0.149}\left(\frac{\eta_c}{\eta_{water}}\right)$ $C_{Grace,1} = 0.94{C_{Grace,2}}^{-0.757}\ (2 < C_{Grace,2} < 59.3)$ $C_{Grace,1} = 3.42{C_{Grace,2}}^{-0.441}\ (C_{Grace,2} > 59.3)$ $\boldsymbol{V_\infty} = \frac{\boldsymbol{\eta_c}}{\boldsymbol{d\rho_c}}\boldsymbol{Mo}^{-0.149}\left(\boldsymbol{C_{Grace,1}} - \mathbf{0.875}\right)$
Henschke (2004)	$\mathbf{V}_\infty = \left(\frac{\mathbf{V_{sph}V_{nsph}}}{\mathbf{V_{sph}}^{\boldsymbol{a_{16}}} + \mathbf{V_{nsph}}^{\boldsymbol{a_{16}}}}\right)^{\frac{1}{\boldsymbol{a_{16}}}}$

3.1.2. Henschke model

- Combine different models

$$V_{\infty,\,Sph} = \frac{Re_{\infty,Sph}\eta_c}{\rho_c d}$$

$$V_{\infty,Osc} = \sqrt{\frac{2a_{15}\sigma}{\rho_c d}}$$

Maneri (1995)

$$V_{\infty,\,Def} = \sqrt{\frac{\Delta\rho g d}{2\rho_c}}$$

Clift et al. (1978)

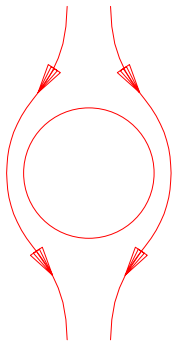
- Use crossing-over functions and fitting parameters to adjust them and cover all the drops diameter range;

- Takes in account the dispersed phase viscosity.

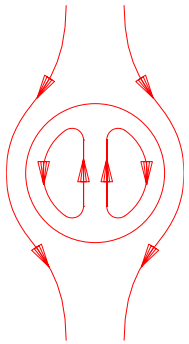
3.1.3. Henschke model

Spherical drop

rigid drop

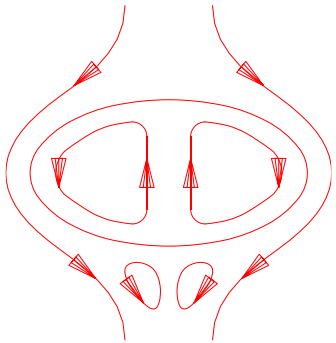


circulating drop

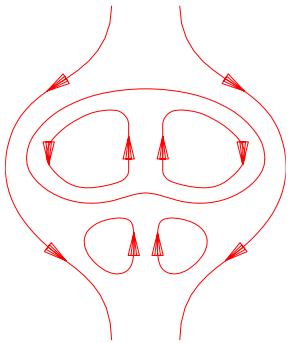


Non Spherical drop

oscillating drop



deformed drop



M. Henschke, (2003)

3.1.3. Henschke model

Rigid spherical drops:

$$Ar = \frac{\rho_c(\rho_c - \rho_d) g d^3}{\eta_c^2}$$

$$C_w = \frac{432}{Ar} + \frac{20}{Ar^{\frac{1}{3}}} + \frac{0.51 * Ar^{\frac{1}{3}}}{140 + Ar^{\frac{1}{3}}}$$

$$Re_{\infty,\, rig} = \sqrt{\frac{4Ar}{3Cw}}$$

$$Re = \frac{\rho_d * V * d}{\eta_c}$$

$$V_{\infty,\, rig} = \frac{\eta_c * Re_{\infty,\, rig}}{\rho_d * d}$$

3.1.3. Henschke model

Circulating drops:

$$0 < Re_{\infty,Circ} < 500$$

$$Re_{\infty,Circ} = \frac{Ar}{a_{Da}(0.065Ar + 1)^{1/6}}$$

$$V_{\infty,circ} = \frac{\eta_c * Re_{\infty,Circ}}{\rho_d * d}$$

Spherical drops (rigid and circulating):

$$f_2 = 1 - \frac{1}{1 + \left(\frac{d}{d_{sw}}\right)^{a_{sw}}}$$

$$K'_{HR} = \frac{3\left(\eta_c + \frac{\eta_d}{f_2}\right)}{2\eta_c + 3\frac{\eta_d}{f_2}}$$

3.1.3. Henschke model

Spherical drops:

$$f'_1 = 2 * (K'_{HR} - 1)$$

$$Re_{\infty,\,Sph} = \left(1 - f'_1\right) * Re_{\infty,\,Rig} + f'_1 * Re_{\infty,\,Cir}$$

$$V_{\infty,\,spherical} = \frac{\eta_c * Re_{\infty,\,sph}}{\rho_d * d}$$

3.1.3. Henschke model

Oscillating drops:

$$V_{\infty,\,Osc} = \sqrt{\frac{2 * a_{15} * \sigma}{\rho_d * d}}$$

Deformed drops:

$$V_{\infty,\,Def} = \sqrt{\frac{\Delta\rho * g * d}{2 * \rho_c}}$$

Non spherical drops:

$$V_{\infty,nSph} = \left(V_{os}^{a_{gr}} + V_{def}^{a_{gr}}\right)^{\frac{1}{a_{gr}}}$$

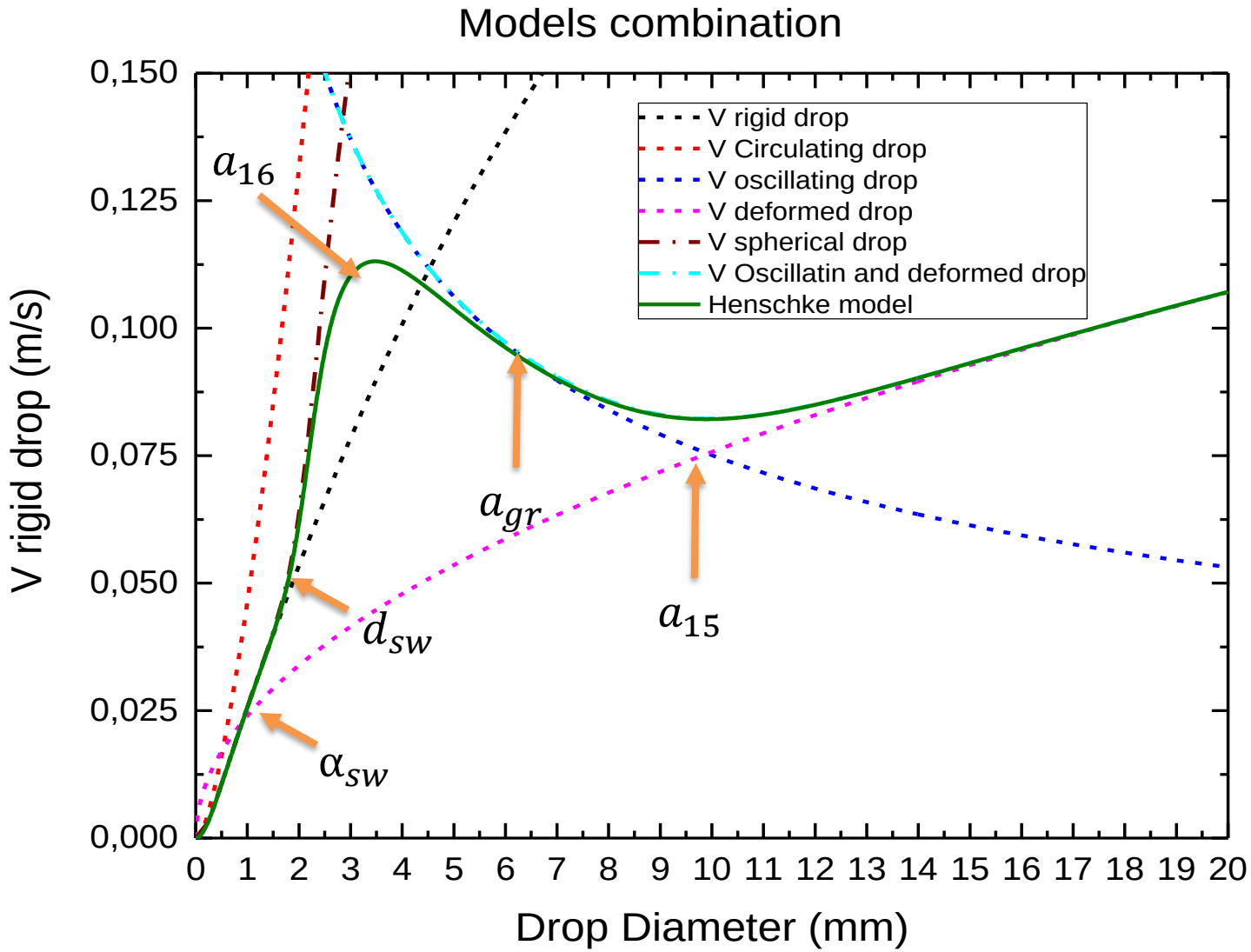
Henschke model:

$$V_\infty = \left(\frac{V_{\infty,\,Sph} * V_{\infty,\,nSph}}{V_{\infty,\,Sph}^{a_{16}} + V_{\infty,nSph}^{a_{16}}}\right)^{\frac{1}{a_{16}}}$$

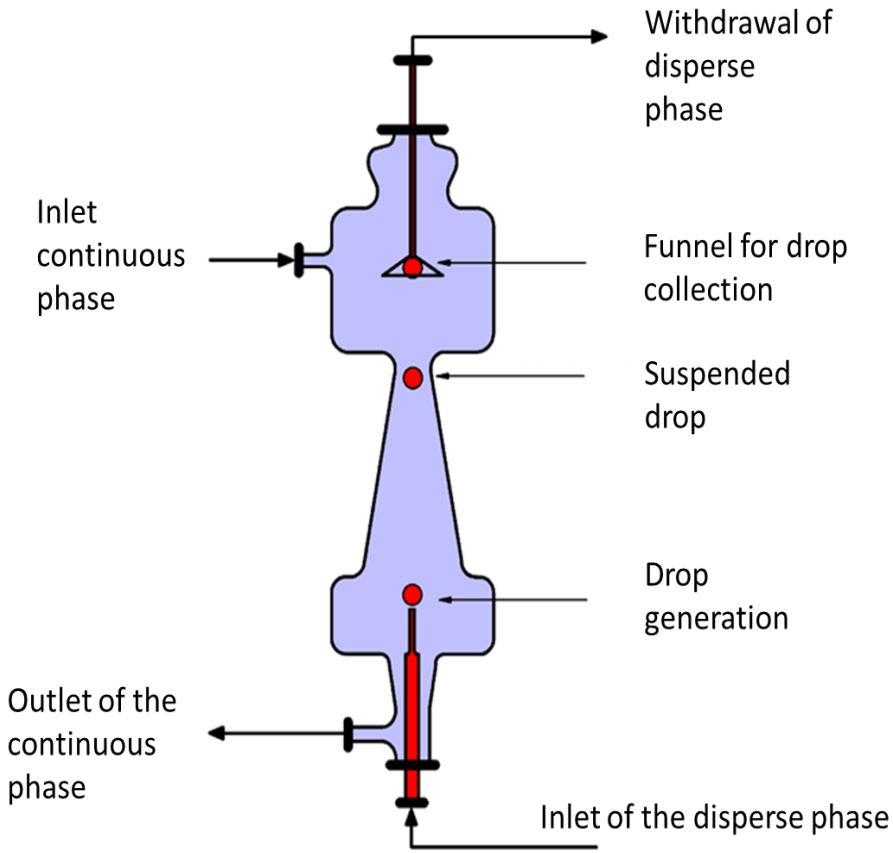
3.1.3. Henschke model

Fitting parameters	
a_{DA}	Adjustement to the real flowing behavior of circulating drops
d_{sw} (mm)	Transition drop diameter between rigid and mobile interface
a_{sw}	Scales the steepness of the crossover within the spherical drop domain
a_{15}	intensity and effect of oscillation
a_{gr}	Transition within the non-spherical drops domain
a_{16}	smoothness of transition all over the range

3.1.3. Henschke model



3.2. Mass transfer experiments



- Lab scale single drops experiments at different viscosities for both phases ;
- Model validation;
- Integration of the results in ReDROP

3.2.1. Mass Transfert modelling

- Goal:
  - Overall molar flux derived from the 2 films theory

$$\dot{M} = K_{od}A\left(C_y^* - C_y\right) = K_{oc}A(C_x - C_x^*)$$

$$\frac{1}{K_{od}} = \frac{1}{k_d} + \frac{P}{k_c}$$

  - Overall transfer coefficient

or

$$\frac{1}{K_{oc}} = \frac{1}{k_c} + \frac{1}{P * k_d}$$

3.2.1. Mass Transfert modelling

- Target:
  - mass-transfer coefficient k
  - diffusion coefficient D

$$Sh = \frac{k * d}{D} \qquad Sc = \frac{\eta}{\rho * D} \qquad Pe = Re * Sc$$

3.2.2. Continuos phase

Calculation of the mass-transfer coefficient k

$$Sh_c = \mathrm{cst} + C_1 Re^{C_2} Sc^{C_3}$$

Garner *et al.* (1958); Garner *et al.* (1959); Steiner (1986); Baird and Hamielec (1962); Lochiel and Calderbank (1964); Garner and Tayeban (1960); Thorsen and Terjesen (1962).

3.2.3. Disperse phase

- The considerable smaller volume of the dispersed phase (single drop);
- The different hydrodynamic patterns inside a drop;
- The short time a drop spend in a column;
- Time dependent process.

Fourier number

$$Fo_d = \frac{4 * D_d * t}{d^2}$$

3.2.3. Disperse phase

Kronik and Brink (1950) $$D_{eff} = R * D_d$$ R= 2.5

Handlos and Baron (1957) $$D_{eff} = \frac{V_\infty * d}{2048 * \left(1 + \frac{\eta_d}{\eta_c}\right)}$$

Henschke (2004) $$D_{eff} = D_d + \frac{V_\infty * d}{C_{IP} * \left(1 + \frac{\eta_d}{\eta_c}\right)}$$

Kalvoda (2016) $$D_{eff} = D_{phys} + \frac{V_\infty}{C_{prof} * \left(1 + \frac{\eta_d}{\eta_c}\right)}$$

3.2.3. Disperse phase

Wilke and Chang (1955)

$$D_d = 7.4 \times 10^{-8} * \frac{T * (\varphi M)^{\frac{1}{2}}}{\eta * V^{0.6}} \quad \text{with} \quad \varphi M = \sum_{\substack{j=1 \\ j \neq A}}^{n} X_j * \varphi_j * M_j$$

Perkins and Geankoplis (1969)

$$D_d * \eta_m^{0.8} = \sum_{\substack{j=1 \\ j \neq A}}^{n} X_j * D_{d,j} * \eta_j^{0.8}$$

3.2.3. Disperse phase

A fractional approach to equilibrium: dimensionless concentration ratio (E or y^+)

$$E = \frac{y(t) - y^*}{y_0 - y^*}$$

Newman (1931): $$E = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp(-(n * \pi)^2 * Fo_d)$$

The mass-transfer coefficient is calculated:

$$Sh_d = -\frac{d^2}{6 * D_d * t} \ln \left[\frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp(-(n * \pi)^2 * Fo_d) \right]$$

3.2.3. Disperse phase

Mersmann (1986)

Garthe (2006)

For short contact times

$$Fo_d < 0.1584 \qquad E = 1 - 6 * \sqrt{\frac{Fo_d}{\pi}} + 2.996 * Fo_d \qquad k_d = \sqrt{\frac{D_d}{\pi * t}}$$

For long contact times

$$Fo_d \geq 0.1584 \qquad E = \frac{6}{\pi^2} exp[-\pi^2 * Fo_d] \qquad k_d = \frac{2 * \pi^2 * D_d}{3 * d}$$

4. Programming aspect

- Rewrite ReDrop:
  - Translate it from Cpp to Fortran;
  - Combine all the versions taking in account different aspect;
  - Integrate the result of the present study;
  - Add an optimization tool.
- Test and validate it at industrial scale.

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