



Online *in situ* viscosity determination in stirred tank reactors by measurement of the heat transfer capacity

Martin Wunderlich^a, Patrick P. Trampnau^a, Emanuel F. Lopes^b, Jochen Büchs^{a,*}, Lars Regestein^a

^a RWTH Aachen University, AVT – Aachener Verfahrenstechnik, Biochemical Engineering, Worringer Weg 1, 52074 Aachen, Germany

^b Instituto Superior Técnico, Universidade de Lisboa, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

HIGHLIGHTS

- Online and *in situ* viscosity determination in turbulent stirred tank reactor.
- Viscosity determination is based on the measurement of the heat transfer capacity.
- Precise measurement of heat transfer capacity by means of a calibration heater.
- Viscosity determination showed to be precise and accurate.

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ABSTRACT

Viscosity plays an important role in a variety of biotechnological and chemical processes, such as in the production of biopolymers and in fermentations with filamentous microorganisms as well as in some dissolution, crystallization, and hydrogenation processes. Most of the established online methods for measuring the viscosity, however, struggle with the complexity of multiphase liquids like aerated liquids or suspensions and fermentation broths. This work presents a method to consider the viscosity of the whole reactor content regardless of its composition by means of calorimetric measurement of the heat transfer capacity (UA) and the use of a heat transfer model. Measurements were carried out with polyvinylpyrrolidone (PVP) model solutions (0–110 g/L) in a 50 L pilot scale stirred tank reactor with different viscosities (0.001–0.12 Pa·s), mechanical power input (0.04–27 kW/m³), and aeration rates (0–2 vvm). The heat transfer capacity (UA) measurement by means of a calibration heater (1.25–12.5 kW/m³) was found to be very precise (< 1.5% standard deviation) and the online *in situ* determination of the viscosity fairly accurate (9.8% arithmetic mean error) in comparison to the offline measured viscosity. This suggests that the presented method is suited for online *in situ* viscosity determination in stirred tank reactors.

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1. Introduction

Viscosity is a key parameter in various fermentation processes like in the production of extracellular polysaccharides like xanthan, alginate, pullulan, or polyglutamic acid as well as in fermentations with filamentous biomass growth, as observed for many fungi and *Streptomyces sp.*, e. g. during the production of antibiotics and organic acids (Henzler, 2007). In the chemical industry, unit operations as hydrogenation (e.g. of vegetable oils), crystallization, and dissolution (Nienow, 2000) may show notable

viscosity changes. Therefore, the viscosity is often directly linked to product formation and biomass growth, respectively, and has direct impact on the mixing performance and generally on mass transfer (Henzler, 2007). Consequently, the ability to measure the viscosity inside a stirred tank reactor (STR) is of special interest for online monitoring and process control.

1.1. Methods for measuring the viscosity

Until some 25 years ago, viscosity measurement was commonly carried out with offline samples using, for instance, a rheometer for a detailed analysis of the rheological behavior of the sample. Though offline measurement provides detailed

* Corresponding author.

E-mail address: jochen.buechs@avt.rwth-aachen.de (J. Büchs).

Nomenclature*Latin letters*

A	Heat exchange area (0.424 m^2)
a	Empirical exponent of Reynolds number, Eq. (8).
B	Rheology factor, Eq. (21).
b	Empirical exponent of Prandtl number, Eq. (8).
C_r	Constant accounting for the geometry of reactor and stirrer, Eq. (8).
$c_{p,r}$	Heat capacity at constant pressure of vessel liquid ($30 \text{ }^\circ\text{C}$)* ($4180 \text{ kJ kg}^{-1} \text{ K}^{-1}$)
$c_{p,j}(T_j)$	Heat capacity at constant pressure of jacket liquid** [$\text{kJ kg}^{-1} \text{ K}^{-1}$]
D_O	Inner diameter of jacket inlet (0.025 m)
D_R	Inner diameter of reactor vessel (0.29 m)
D_{str}	Stirrer diameter (0.12 m)
d	Characteristic dimension [m]
d_s	Characteristic dimension of jacket, $d_s = (8/3)^{0.5} \cdot \delta$ [m]
e	Empirical exponent of viscosity number, Eq. (8).
F_{gas}	Volume specific aeration rate (at $0 \text{ }^\circ\text{C}$, 101325 Pa) [$\text{m}^3_{gas} \text{ m}^{-3} \text{ min}^{-1}$]
Fr	Froude number, $Fr = n^2 \cdot D_{str}/g$ [-]
g	Gravitational acceleration of the earth (9.81 m s^{-2})
Ga	Galileo number, $Ga = D_R^2 \cdot g \cdot (\eta_n/\rho_L)^{-2}$ [-]
$h_{L,0}$	Height of unaerated liquid in vessel (0.625 m)
h_e	Distance between reactor bottom and sparger (0.002 m)
h_j	Height of jacket cylinder (0.468 m)
K	Consistency factor [$\text{Pa} \cdot \text{s}^m$]
L	Adjustment parameter for shear rate calculation, see (Henzler, 2007), Eqs. (20), (22).
l_j	Mean path length through jacket (1.21 m)
m	Flow behavior index [-]
\dot{m}_j	Mass flow rate through jacket [kg s^{-1}]
$M_{ln}(T_1, T_2)$	Logarithmic mean value of T_1 and T_2 [K]
n	Stirring rate [s^{-1}]
Nu	Nusselt number [-]
P_{str}	Mechanical power input of stirrer [kW]
Po_G	Gassed power number, $Po_G = P_{str,G}/(\rho_L \cdot n^3 \cdot D_R^5)$ [-]
Pr	Prandtl number [-]
\dot{Q}_{cal}	Calibration heat flow [W]
q_{cal}	Volume specific calibration heat flow [kW m^{-3}]
R^2	Coefficient of determination [-]
R_f	Thermal resistance due to fouling [$\text{m}^2 \text{ K W}^{-1}$]
Re	Reynolds number [-]
Re_G	Gas Reynolds number [-]
s	Wall thickness of inner reactor wall (0.005 m)
T_r	Reactor temperature [K]
$T_{j,in}$	Jacket inlet temperature [K]
$T_{j,out}$	Jacket outlet temperature [K]

u_a	Water velocity in annular space of jacket [m s^{-1}]
u_b	Water velocity in jacket due to buoyancy [m s^{-1}]
u_j	Mean water velocity in jacket [m s^{-1}]
u_o	Water velocity in inlet tube of jacket [m s^{-1}]
U	Overall heat transfer coefficient [$\text{W m}^{-2} \text{ K}^{-1}$]
UA	Heat transfer capacity [W K^{-1}]
UA_{meas}	Measured heat transfer capacity [W K^{-1}]
$V_{r,L}$	Liquid volume of reactor (0.04 m^3)
$V_{j,L}$	Liquid volume of jacket (0.00377 m^3)

Greek letters

α	Convective heat transfer coefficient [W K^{-1}]
$\beta_{j,L}(T_j)$	Coefficient of volumetric thermal expansion** [W K^{-1}]
$\dot{\gamma}_{r,n}$	Shear rate in vessel bulk [s^{-1}]
δ	Reactor annulus width (0.00795 m)
$\eta_{r,n}$	Apparent dynamic viscosity of vessel bulk [$\text{Pa} \cdot \text{s}$]
$\eta_{j,n}$	Apparent dynamic viscosity of jacketbulk*** [$\text{Pa} \cdot \text{s}$]
$\lambda_{r,L}$	Thermal conductivity of reactor liquid* ($0.614 \text{ W m}^{-1} \text{ K}^{-1}$)
$\lambda_{j,L}(T_j)$	Thermal conductivity of jacket liquid** ($\text{W m}^{-1} \text{ K}^{-1}$)
λ_{steel}	Thermal conductivity of stainless steel 1.4435 ($15 \text{ W m}^{-1} \text{ K}^{-1}$)
ξ	Energy dissipation parameter of stirrer (0.285, interpolated; Kurpiers, 1985)
$\rho_{r,L}$	Mass density of reactor liquid ($30 \text{ }^\circ\text{C}$)* (995.65 kg m^{-3})
$\rho_{j,L}(T_j)$	Mass density of jacket liquid** [kg m^{-3}]
τ	Residence time of circuit water in jacket [s]
*	Exact values are unknown and, therefore, approximated with the values of water at $30 \text{ }^\circ\text{C}$ given by (Wagner and Kretschmar, 2010).
/	Exact values for water were calculated with the logarithmic mean temperature of the jacket (T_j) with a polynomial regression function. The Polynomial were of 2nd (**) or 3 rd (***) degree and fitted to the data between $10 \text{ }^\circ\text{C}$ and $40 \text{ }^\circ\text{C}$ given by Wagner and Kretschmar (2010).

Indices

i	Counter for sum
j	Jacket side
L	Liquid
\max	Maximum
n	In bulk
off	When calibration heater is off
on	When calibration heater is on
r	Reactor side
w	In wall boundary layer

information about the rheology of a sample, the sample itself may not be representative for certain processes: When separating a sample from the bulk, conformity changes may occur due to gelation, thixotropic behavior, phase separation of emulsions and suspensions, or mechanical damage due to the sampling itself as for example because of high shear in a sampling valve (Chhabra and Richardson, 2008). Furthermore, for control of the product quality and of the production process, for process automation and for cost reduction, online viscometry was established (Chhabra and Richardson, 2008).

For online viscosity measurement there are different approaches: In stirred tank reactors, the mechanical power input of the stirrer can be measured for determining the viscosity (impeller viscometer) (Doran, 2013; Morawski et al., 1999). However, this method is limited to the laminar flow regime or to non-baffled STRs when using standard (fast rotating) impellers such as Rushton or pitched blade turbines, or marine propellers. Only under these conditions the power number changes with the Reynolds number and, therefore, with the viscosity. But, in order to provide homogeneous mixing and a sufficient oxygen supply for aerobic

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