CHEMICAL ENGINEERING TRANSACTIONS
VOL. 74, 2019
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.cetjournal.it
Guest Editors: Sauro Pierucci, Jiří Jaromír Klemeš, Laura Piazza

ISBN 978-88-95608-71-6; **ISSN** 2283-9216
DOI: 10.3303/CET1974225

Comparative Study on Calorimetric Determination of Power Numbers in a Lab Scale Batch Reactor

Lennart Camps[a]*, Luc Moens[b], Urs Groth[c], Leen Braeken[a], Simon Kuhn[d], Leen C.J. Thomassen[a]

[a]KU Leuven, Faculty of Engineering Technology, Lab4U; Agoralaan building B box 8, 3590 Diepenbeek,Belgium
[b]Janssen Pharmaceutica, Turnhoutseweg 30, 2430 Beerse,Belgium
[c]Mettler Toledo GmbH, Heuwinkelstrasse 3, 8606 Nänikon, Switzerland
[d]KU Leuven, Department of Chemical Engineering, Celestijnenlaan 200F, 3001 Leuven,Belgium
lennart.camps@kuleuven.be

Accurate measurement of power numbers on lab scale reactors can provide useful information about the needed energy to reduce mass transfer problems. Next, this knowledge can be used to avoid scale-up problems (Paul et al., 2004). In this study, the accuracy and applicability of four methods to determine the power number in a 1 L reactor are tested: calorimetry, torque measurement, computational fluid dynamics (CFD) and the Furukawa et al. correlation (Furukawa et al., 2012). Experiments with water and 2-octanol are performed with a 4-bladed 45 ° pitched blade turbine (4PBT) and 6-bladed Rushton turbine in baffled and unbaffled conditions. At low rotational speed, experimental techniques record higher power numbers compared to the theoretical techniques. The calorimetric method is only accurate at rotational speeds above 300 RPM due to insufficient heat flow at lower settings. Torque measurement is most accurate at low rotational speed, before the vortex reaches the stirrer and creates cavities that lead to inaccurate results. At rotational speeds higher than 300 RPM, differences between the four techniques are less than 30 %, confirming the accuracy of all methods. Therefore, literature correlations can be used for a quick estimation of the power number in lab scale reactor experiments at high rotational speed. However, experimental techniques are recommended for accurate power number measurements at low rotational speed and for specific reactor set-ups which are not described in literature.

1. Introduction

Insufficient mixing and mass transfer reduce reaction rate and impact product distribution, resulting in typical yield losses of 5 %, which have led to a cost of at least $1 billion in the U.S. chemical industry (Paul et al., 2004). These losses mainly occur due to errors during scale-up, where inaccurate measurements at lab scale lead to insufficient mixing at industrial scale. Therefore, this comparative study investigates methods to determine power numbers on lab scale, since power consumption and power density are the most important factors to determine mixing performance at different scales (Furukawa et al., 2012; Paul et al., 2004). For both parameters, the power number, shown in Eq(1), plays a crucial role (Scargiali et al., 2013).

$$N_p = \frac{P}{\rho * N^3 * D^5} \quad (1)$$

The power number, N_p, is related to the electrical power, P, the density, ρ, the rotational speed, N, and the stirrer diameter, D. The Handbook of industrial mixing defines the power number as: “a dimensionless parameter that provides a measure of the power requirements for the operation of an impeller” (Paul et al., 2004). At industrial scale, power numbers are accurately measured, since the power consumed by the stirrer and the dimensions of the vessel are a magnitude larger compared to lab scale. At lab scale, measurement of power numbers is not straightforward and less accurate. Therefore, this study investigates the accuracy of four different techniques to measure power numbers in a 1 L batch reactor.

Paper Received: 18 May 2018; Revised: 11 November 2018; Accepted: 27 April 2019

Please cite this article as: Camps L., Moens L., Groth U., Braeken L., Kuhn S., Thomassen L., 2019, Comparative Study on Calorimetric Determination of Power Numbers in a Lab Scale Batch Reactor, Chemical Engineering Transactions, 74, 1345-1350
DOI:10.3303/CET1974225

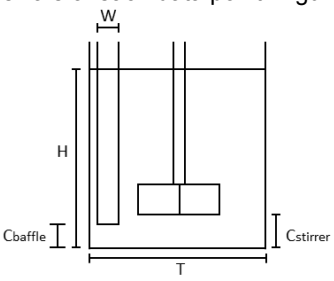
Several methods exist to determine the power number of a stirred batch reactor (Ascanio et al., 2013). The methods can be divided into two main groups: experimental techniques and theoretical methods, as shown in table 1. The most commonly used method for the determination of power numbers in industrial vessels is torque measurement (Kresta et al., 2016). However, as the scale of the reactor decreases, the accuracy of the torque measurement decreases as well, because the torque meter measures both power consumed by fluid mixing and power losses at mechanical parts of the rotor (Ascanio et al., 2013; Cortada-Garcia et al., 2017).
Since all energy added to the fluid by the stirrer dissipates as heat, power number measurements using calorimetry are possible (Ascanio et al., 2013; Paul et al., 2004). Since adiabatic conditions and thus no net heat exchange between reactor and environment are required to accurately measure power numbers by calorimetry, this method is only suitable at lab scale. Computational fluid dynamics (CFD) is a theoretical tool that determines power numbers at various reactor scales. Since this technique is solely based on equations and modelling, a comparison with an actual measurement is needed to validate the computations on each scale (Cortada-Garcia et al., 2017). Similarly, literature correlations need a validation in order to evaluate the effectiveness of the equation for the specific set-up. Besides, empirical correlations are only applicable if a reactor set-up is in good agreement with the reactor set-up used for the derivation of the correlation.

Table 1: Comparison between power number measurement technique

	Experimental				Theoretical			
Techniques		Torque		Calorimetry		Computational fluid dynamics (CFD)		Empirical correlations
Advantages	+	Uncomplicated	+	Lab scale	+	On all scales	+	Immediate
	+	In situ	+	In situ			+	No experimental work
	+	Fast						
Drawbacks	–	Accuracy decreases at lab scale	–	Adiabatic conditions needed	–	Assumptions	–	No in situ measurement
					–	No in situ measurement	–	Limited to reactor set-up

2. Materials and methods

For all experiments, the OptiMax of Mettler Toledo is used. The reactor of 1 L is filled with 800 mL of fluid to perform the experiments. Experiments are performed without baffle, unless indicated otherwise. The four different methods are compared by the average error which is calculated by taking the average of the relative errors of each data point. Figure 1 shows the reactor and the corresponding dimensions.



Part	Symbol	Dimension (mm)
Stirrer	Cstirrer	15
Tank	T	99
	H	95
Baffle	Cbaffle	7
	W	15

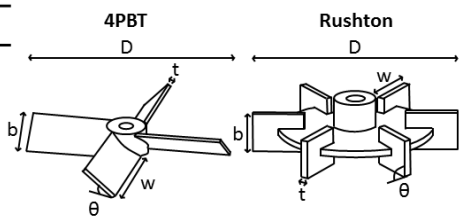


Figure 1: Reactor set-up and details of the used impellers.

For the comparative study, two types of stirrers are used, a 4-bladed 45 ° downward pitched blade turbine (4PBT) and a 6-bladed Rushton turbine, which are shown in Figure 1 and their details are specified in Table 2. Table 3 shows the used solvents with corresponding solvent characteristics.

Table 2: Geometries of the used stirrers

Stirrer	Diameter D (mm)	Blade height b (mm)	Blade width w (mm)	Blades	Blade thickness t (mm)	Angle θ (°)
4PBT	46	24	16	4	1.5	45
Rushton	46	17	10	6	1.5	90

Table 3: Solvent characteristics at 20°C

Solvent	Company	Purity	Viscosity (mPa.s)	Density (kg/m³)
Water	/	Ultrapure (Cond. ~ 0.055 µS/cm)	1.0	998
2-octanol	Sigma-Aldrich	97 %	6.2	821

2.1 Calorimetry

The calorimetric lab tests are performed at a reactor temperature of 20 °C and at a constant rotational speed. Secondly, the overall heat transfer coefficient, U, and the heat transfer surface area, A, are automatically defined using the OptiMax HFCal module (Mettler Toledo) and iControl 5.2 software (Mettler Toledo). Thirdly, the average temperature difference between jacket (T_j) and reactor (T_r) is calculated at a reactor temperature of 20 °C. Next, the heat flow (Q_{flow}) from the reactor to the jacket is determined using Eq(2).

$$Q_{flow} = UA*(T_r-T_j) \quad (2)$$

The heat flow is the net amount of heat the jacket needs to remove from the reactor in order to keep the temperature constant. Figure 2 shows the heat flows at multiple rotational speeds.

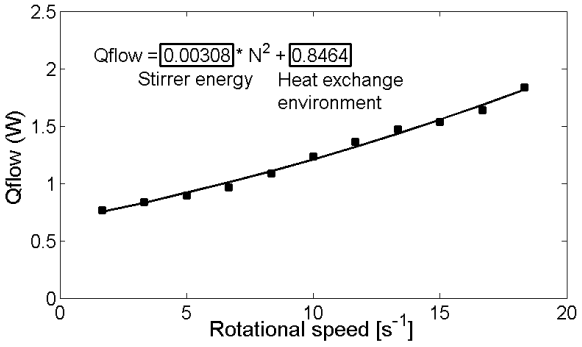


Figure 2: Influence of rotational speed on the heat flow for a 4PBT in water

The increase in heat flow from reactor to jacket at increasing rotational speed is shown in figure 2. An increase in stirrer energy results in an increase in energy dissipated as heat and thus a larger heat flow. Since the room temperature remains approximately constant and is higher than the reactor temperature, there is a constant offset at the heat flow which corresponds to the heat added by the environment. By determining the heat flow at multiple rotational speeds, the contribution of heat from the environment can be separated from the heat caused by the energy dissipation of the stirrer. The stirrer heat equals the stirrer power and therefore Eq(1) is used to calculate the power number.

2.2 Torque

iControl 5.2 software is used to perform experiments with the OptiMax lab reactor. Dependent on the reactor set-up, the torque (RT) of the automated lab reactor is monitored in two different ways: via the conventional stirrer drive or magnetic drive (MAGdrive) of the OptiMax. The MAGdrive drives the stirrer shaft frictionless, where the coupling of motor and stirrer is ensured by two physically separated magnets. The conventional stirrer drive is directly connected to the shaft. The friction is neither constant nor defined. MAGdrive has less mechanical power losses and is therefore assumed to be more accurate than conventional stirrer drive (Cortada-Garcia et al., 2017; Paul et al., 2004). The average torque at each rotational speed is measured by applying a ramp from 100 to 800 RPM at 5 RPM/min. The power of the stirrer at a rotational speed is calculated with the measured torque using Eq(3) (Major-Godlewska and Karcz, 2017; Tamburini et al., 2016). Eq(3) then translates the power into the power number.

$$P = 2*\pi*N*RT \quad (3)$$

2.3 Furukawa power number correlation

In literature, many studies propose correlations to predict power numbers in batch reactors (Furukawa et al., 2012; Paul et al., 2004; Scargiali et al., 2013). Furukawa et al. developed correlations for lab scale batch reactors (Furukawa et al., 2012). The correlations are stirrer dependent and include the Rushton turbine and 4PBT. Since the same reactor geometry ratios and stirrers are used in this research, the existing correlations can be applied.

2.4 CFD

The software used for CFD is Comsol Multiphysics® 4.4. The calculation of the power number is performed by integrating the power draw over the surface of the stirrer and stirrer shaft in the rotating machinery mixer module (Min and Gao 2006; Paul et al. 2004). The modelling uses a RANS (Reynolds-averaged-Navier-Stokes) k-ε (kinetic energy-energy dissipation) turbulence model to determine the power numbers. No-slip boundary conditions are used for all solid surfaces. A normal sized tetrahedral mesh is applied to the volume. Newton's method for solving a non-linear system of equations is used as stationary solver, where a solution is found when the relative tolerance is below 10^{-5}. A GMRES (generalized minimal residual) solver obtains the solution for each iteration.

3. Results and discussion

The power numbers for the four techniques using an unbaffled batch reactor with 800 mL of water with a Rushton turbine, are presented in Figure 3. For calorimetry at low rotational speed, the heat flow due to mixing compared to the total heat flow is very low, resulting in large uncertainties in power numbers. Therefore, accurate measurements by calorimetry can only be performed at a rotational speed above 300 RPM (Re > 10^4), which is the standard application range at lab scale. Deviations on torque measurement are negligible (< 1 %) and therefore not added.

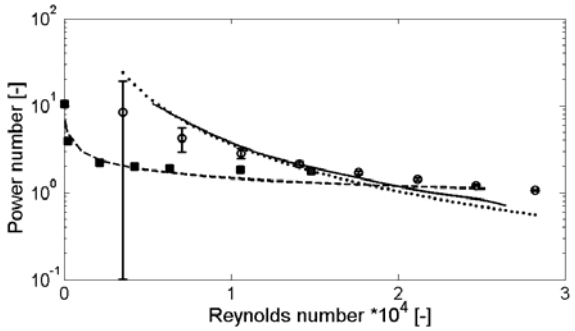


Figure 3: Comparison between calorimetry (○), MAGdrive (—) and conventional torquemeter (• •), Furukawa et al. correlation (– –) and CFD (■) as power number measurement techniques for a Rushton turbine in water

Figure 3 shows that the two torque measurement techniques result in similar power numbers, with an average error of 10 %. Therefore, in the next graphs, only one torque measurement technique is shown. The experimental techniques have a similar trend, where the average difference between calorimetry and MAGdrive torque measurement is 25 %. The theoretical techniques are comparable with an average difference of 18 %. At Re > 10^4, thus at a high rotational speed (N > 300 RPM), all four methods have similar values, 0.9 ± 0.3. However, it should be noted that the torque measurements differ from the other methods at Re > $2*10^4$ as the vortex reaches the stirrer and creates cavities behind the stirrer blades, resulting in deviations on the torque measurement (Scargiali et al., 2013). The determined power numbers correspond to the literature power numbers of 0.9 for a Rushton turbine in an unbaffled batch reactor (Assirelli et al., 2008). The average error of 78 % at low Reynolds numbers (Re < 10^4) between theoretical techniques and torque measurement, indicates that theoretical methods are less suitable to determine power numbers at low Reynolds numbers.

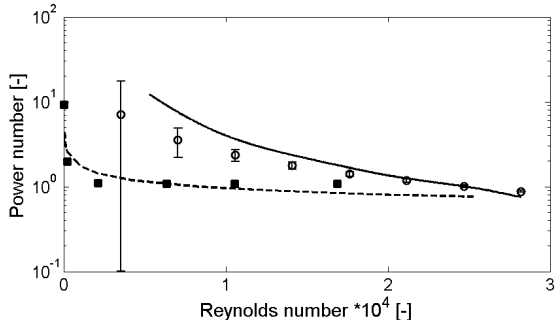


Figure 4: Comparison between calorimetry (○), MAGdrive torquemeter (—), Furukawa et al. correlation (– –) and CFD (■) as power number measurement techniques for a 4PBT in water

Figure 4 shows the power number measurements for the 4PBT in water. An average error of 20 % occurs between the experimental techniques, while the average error between the theoretical techniques is 24 %. All techniques have power numbers of 0.9 ± 0.2 at Re > 10^4 (N > 300 RPM). The power numbers of the experimental techniques are 80 % higher compared to the theoretical techniques at low Reynolds numbers. The trend of the MAGdrive torquemeter starts to deviate at Re > 2.6 * 10^4, where the vortex reaches the stirrer and reduces the power number by creating cavities behind the stirrer blades (Scargiali et al., 2013). This makes the method less interesting to use once the vortex reaches the stirrer. Figure 5 shows the power numbers where 800 mL of 2-octanol is used as solvent.

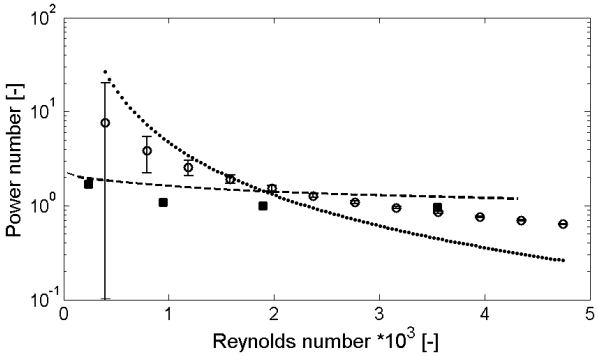


Figure 5: Comparison between calorimetry (○), conventional torquemeter (• •), Furukawa et al. correlation (– –) and CFD (■) as power number measurement techniques for a 4PBT in 2-octanol

Important in Figure 5 is that the Reynolds numbers are a factor 10 lower than the previous figures due to the increased viscosity and decreased density of 2-octanol. Due to reactor set-up limitations, these experiments are only carried out with the conventional stirrer drive. Theoretical techniques have an average error of 24 %, however, the experimental techniques differ from each other, resulting in an average error of 40 %. The differences between torque measurement and the other techniques are up to 70 % at Re > 4*10^3, and are presumably caused by the formation of a large vortex (Scargiali et al., 2013). In contrast to torque measurement, differences between CFD, literature and calorimetry are below 20 % at Re > 2*10^3 (N > 300 RPM). Figure 6 studies the impact of one baffle on the power number for a 4PBT with 800 mL of water.
Similar to the experiments without baffle, two groups can be observed in figure 6: experimental techniques and the theoretical techniques. All four methods have a power number value around 1.0 ± 0.4 at Re > 10^4. This is similar to the unbaffled experiments. In contrast to industrial scale, the influence of the baffle on the power number is limited on a lab scale reactor (Paul et al., 2004).

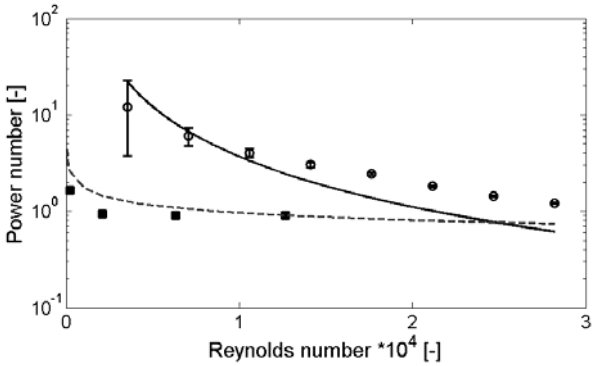


Figure 6: Comparison between calorimetry (○), MAGdrive torquemeter (—), Furukawa et al. correlation (– –) and CFD (■) as power number measurement techniques for a baffled reactor with a 4PBT in water

4. Conclusion

This paper studies the accuracy of four different techniques to measure power numbers in a 1 L batch reactor. An accurate value of the power number is an important first step towards a correct scale-up. The results of the four different methods indicate two different groups: the theoretical values and the practical values. In general, at a rotational speed above 300 RPM in the unbaffled reactor, power numbers of the four methods have

differences below 30 %, but at lower rotational speed, a difference exists between the experimental and theoretical group. Since the experimental techniques effectively measure the energy of the stirrer transferred to the liquid, these techniques are expected to be more accurate for measuring power numbers at low rotational speed.

The calorimetric method has a reduced sensitivity at low rotational speeds, where the stirrer energy added to the reactor volume is small, resulting in large uncertainties on the results of the power number. Therefore, the calorimetric method is most accurate and applicable at high rotational speed. In contrast to the calorimetric method, the torque measurement is an interesting technique to use at low rotational speed. However, at high rotational speed, power number measurements are less accurate, due to the vortex formation, which creates cavities behind the stirrer blades.

In order to have a rough estimation of the power number over a broad range of rotational speeds, empirical correlations like Furukawa et al. are interesting to use if comparable reactor set-up data exist as they are the fastest method and do not need experimental tests. CFD modelling is less advisable, since this technique is more time-consuming and contains assumptions that need to be validated by experimental measurements. To assess the power number of the reactor set-up more accurately for scale-up, experimental techniques like torque measurement or calorimetry are recommended.

Acknowledgments

This work is made possible by Mettler Toledo and Janssen Pharmaceutica, part of Johnson & Johnson. The authors gratefully acknowledge the financial support of the KU Leuven Impulsfonds IMP/16/023.

References

Ascanio, G., Castro, B., & Galindo, E. (2013). Measurement of Power Consumption in Stirred Vessels—A Review, *82*(September 2004), 1282–1290. https://doi.org/10.1205/cerd.82.9.1282.44164

Assirelli, M., Bujalski, W., Eaglesham, A., & Nienow, A. W. (2008). Macro- and micromixing studies in an unbaffled vessel agitated by a Rushton turbine. *Chemical Engineering Science*, *63*(1), 35–46. https://doi.org/10.1016/j.ces.2007.07.074

Cortada-Garcia, M., Dore, V., Mazzei, L., & Angeli, P. (2017). Experimental and CFD studies of power consumption in the agitation of highly viscous shear thinning fluids. *Chemical Engineering Research and Design*, *119*, 171–182. https://doi.org/10.1016/j.cherd.2017.01.018

Furukawa, H., Kato, Y., Inoue, Y., Kato, T., Tada, Y., & Hashimoto, S. (2012). Correlation of power consumption for several kinds of mixing impellers. *International Journal of Chemical Engineering*, *2012*. https://doi.org/10.1155/2012/106496

Kresta, S. M., Etchels, A. W., Dickey, D. S., & Atiemo-Obeng, V. A. (2016). *Advances in Industrial Mixing: A Companion to the Handbook of Industrial Mixing*. https://doi.org/10.1017/CBO9781107415324.004

Major-Godlewska, M., & Karcz, J. (2017). Power consumption for an agitated vessel equipped with pitched blade turbine and short baffles. *Chemical Papers*, *72*(5), 1081–1088. https://doi.org/10.1007/s11696-017-0346-x

Min, J., & Gao, Z. (2006). Large eddy simulations of mixing time in a stirred tank. *Chinese Journal of Chemical Engineering*, *14*(1), 1–7. https://doi.org/10.1016/S1004-9541(06)60030-X

Paul, E. L., Atiemo-Obeng, V. A., & Kresta, S. M. (2004). *Handbook of Industrial Mixing. Science and Practice*. https://doi.org/10.1002/0471451452

Scargiali, F., Busciglio, A., Grisafi, F., & Brucato, A. (2013). Influence of viscosity on mass transfer performance of unbaffled stirred vessels. *Chemical Engineering Transactions*, *32*, 1483–1488. https://doi.org/10.33032/CET1332248

Scargiali, F., Busciglio, A., Grisafi, F., Tamburini, A., Micale, G., & Brucato, A. (2013). Power consumption in uncovered unbaffled stirred tanks: Influence of the viscosity and flow regime. *Industrial and Engineering Chemistry Research*, *52*(42), 14998–15005. https://doi.org/10.1021/ie402466w

Tamburini, A., Cipollina, A., Scargiali, F., Micale, G., & Brucato, A. (2016). Power Requirements for Complete Suspension and Aeration in an Unbaffled Bioslurry Reactor. *Chemical Engineering Transactions*, *49*, 451–456. https://doi.org/10.3303/CET1649076