Calibration of a Jar-Tester Replicated in a Rotational Rheometer

Emilio Alvarenga¹, Reidar Barfod Schüller² and Carlos Salas-Bringas³

¹Dep. Of Environmental Sciences, Norwegian University of Life Sciences, P.O. Box 5003, N-1432 Ås, Norway.

²Dep. Of Chemistry, Biotechnology and Food Science, Norwegian University of Life Sciences, P.O. Box 5003, N-1432 Ås, Norway.

³Dep. Of Mathematical Sciences and Technology, Norwegian University of Life Sciences, P.O. Box 5003, N-1432 Ås, Norway.

ABSTRACT

A prediction method to determine viscosity in laminar and turbulent regimes was investigated in this article for a replicated Jar-tester adapted to a rotational rheometer. The calibration enables the prediction of an average torque and rotational speed by means of a model for both flow regimes.

INTRODUCTION

Coagulants and flocculants optimal dosage is commonly determined in the wastewater treatment plants (WWTPs) by the Jar-test (JT) device. This instrument resembles a paddle mixer in a cylindrical cup.

In addition, corrective measures can be implemented in the WWTPs for the removal of particles when overflow conditions (e.g. rain water) occur along with re-adjustments for other physicochemical water parameters to keep an efficient separation process that complies with environmental regulations. This fact is firstly attributable to the geometry of the stirrers. Secondly, there are controlled mixing conditions over time in the rapid mixing (coagulation phase) and slow mixing (flocculation phase)¹. Thereby, these type of tests are expected to have a similar flow behavior as in the full plant scale.

Albeit, operators in the plants must consider a theoretical and empirical baseline of the process in order to get close to the real process conditions. Moreover, this particular feature is inherent to every treatment process and thus, iterative calculations are required for matching the baseline conditions and for the achievement of a correct adjustment. Such procedure leads to a time consuming correction in the process¹.

An understanding of the flow behavior is needed due to the changes in viscosity that occur when adding physical or chemical conditioners to the wastewater in a turbulent regime (rapid mixing). Effects associated to turbulence in addition to the viscosity contribution could influence the location of the average shear rate². Conversely, in the laminar regime (slow mixing) the average shear rate could be determined by means of an analogue radius³. The estimation of these average shear rates and average viscosity in both flow regimes would bring additional information about the optimization of coagulants and flocculants when performing standard JT experiments in the WWTPs. Moreover, such approach could provide insight for replicating shear conditions with different geometries for paddles when upscaling similar shear rates in industrial equipment as investigated by Örmeci4.

A calibration procedure is required in order to determine the constants for the determination of the average shear rate and average shear stress like previously studied by Salas-Bringas et al.⁵.

The aim of this study is to determine an average shear rate ($\dot{\gamma}_{avg}$) and its location from rotational speed and an average shear stress (τ_{avg}) from torque for a Newtonian fluid in a replicated Jar-Tester. Both laminar and turbulent regime calibrations are assessed with a mathematical model and experimental data from a rotational rheometer.

MATERIALS AND METHODS

Part 1: Experimental Calibration Procedure

All the measurements for the calibration procedure were carried out with rotational rheometers. The standard fluid utilized was a Newtonian silicon oil labelled as 1000 cP at 25°C (Brookfield Engineering Laboratories, Inc., USA).

The actual viscosity (A_n) of the silicon oil was measured by means of a cone-plate geometry (50 mm diameter, 1°, 50 µm gap) at constant shear rate (5s⁻¹) in an Anton Paar rheometer MCR-301. The temperature range from 20 to 60°C with 10°C ramping steps was taken into consideration for the calibration procedure.

A Jar-tester mixer from a Kemira Flocculator 2000 was replicated in a CAD program, SolidWorks, as seen in Fig. 1 (a) and (b) and printed in an ABS 3D printer (Mojo). The replicated JT was connected to a Paar Physica UDS 200 rheometer as shown in the experimental assembly of Fig. 2. The conditions experimental (speed and temperature) were controlled in order to obtain accurate measurements of the Torque (M) and rotational speed (N). The range from 10 to 50 rpm is used for the slow mixing and from 100 to 500 rpm for the rapid mixing. The temperature range considered for the experiment was from 20 to 60°C with 10°C ramping steps.

The fluid volume was 725 mL and the flow curves (M vs. N) were determined for each temperature in the range of interest in order to calculate two constants K_1 and K_2 . K_1

is used to convert M into an τ_{avg} and K_2 to convert N into an $\dot{\gamma}_{avg}$.





Figure 1: (a) the standard JT (b) 3D drawing of the JT system and its lid and shaft coupled to a Paar Physica UDS200 rheometer



Figure 2: Experimental assembly. The figure shows: (a) a replicate of the JT system assembled in an Anton Paar UDS200 rheometer and (b) the replicated system inside a cylindrical container surrounded with water pumped by a peristaltic pump for the control of the temperature

Part 2: Analytical determination of the average shear stress and mean shear rate for the slow mixing phase

In an analogue cylinder geometry, the τ_{avg} and the $\dot{\gamma}_{avg}$ are given by Eq. 1 and 2 as follows³,

$$\tau = \frac{M}{2\pi r^2 h} \tag{1}$$

$$\dot{\gamma} = \frac{2\pi r N}{60\delta} \tag{2}$$

In these equations, r(m) represents the radius of the analogue cylinder whereas h(m) is its height and $\delta(m)$ is the gap between the stationary wall of the flask and the analogue cylinder. It is thus possible to determine an expression for K_1 and K_2 to obtain η_{avg} . This is shown in Eq. 3, 4 and 5.

$$\eta = \frac{M}{2\pi r^2 h} / \frac{2\pi r N}{60\delta} \tag{3}$$

$$K_1 = \frac{1}{2\pi r^2 h} \tag{4}$$

$$K_2 = \frac{2\pi r}{60\delta} \tag{5}$$

The unit for K_1 is m⁻³ whereas K_2 is a dimensionless number. Eq. 3 can be simplified in order to express the viscosity in terms of a *slope a* that is the ratio between M and N (Nm/rpm) as seen in Eq. 6. Furthermore, δ can be replaced by " $r_{cyl} - r$ " for the iterative calculation of the analogue radius r (m) where r_{cyl} represents the internal radius of the cylindrical container (Fig. 2 (a)). In the JT system, the internal radius r_{cyl} is 0.0445 m and h is 0.0303 m (paddle section).

$$\eta = \frac{15(r_{cyl} - r)}{\pi^2 r^3 h} \cdot slope \ a \tag{6}$$

A relative error (*E*) from the experimental work was calculated in order to determine the accuracy of the experimental measurements of the Newtonian fluid in the calibration procedure, with the coaxial analogue cylinder. The latter predicted viscosity (P_η) obtained by means of the UDS200 rheometer was compared with the actual viscosity (A_{η}) derived from the MCR-301 device. Eq. 7 includes the previously mentioned terms,

$$E = (P_{\eta} - A_{\eta})/A_{\eta} \tag{7}$$

Part 3: Analytical determination of the average shear stress and mean shear rate for the rapid mixing phase

The approach of the linear curve fit was not suitable between M and N for the rapid mixing. In this range of rotational speeds (from 100 to 500 rpm), a turbulent regime was generated and a non-linear trend was hence obtained. It was thus assumed that the τ_{avg} is a function of the η_{avg} and $\dot{\gamma}_{avg}$ as shown in Eq. 8 (in terms of M and N),

$$M = A \cdot N^B \cdot \eta + C \cdot N^D \tag{8}$$

In Eq. 8 η is the A_{η} . It was expressed in terms of the temperature (T, in Kelvin) from the Arrhenius relationship⁶, and it is given by,

$$\eta = e^{(-5.77 + \frac{1722.2}{T})} \tag{9}$$

M is given as μ Nm and *N* as rpm. In order to determine the values of the constants (A and C) and the coefficients (B and D) in Eq. 8, a script was written in MATLAB using the function *fininsearch*⁷ for the optimal minimal value of the function J expressed as,

$$I = \frac{1}{F} \sum (z - \hat{z})^2 \tag{10}$$

Where *F* represents the total set of data points (25) in the space (x, y) and the predicted value, \hat{z} , is given by the Eq. 8 as *M*. Thereby, the function *fminsearch* minimizes the prediction error function J^7 . A relative error distribution for P_η was determined from Eq. 7.

RESULTS AND DISCUSSIONS

Part 1: Determination of the average viscosity in the slow mixing phase

The A_{η} data over the whole temperature range is shown in Table 1.

Table 1: Actual viscosity of the silicon oil measured with a cone plate at $\dot{\gamma} = 5s^{-1}$ in an Anton Paar MCR 301 theometer

AIRON I dai MICKSUT IIICUIIICICI.		
T (°C)	Viscosity (Pa·s)	
20	1.110	
30	0.913	
40	0.759	
50	0.641	
60	0.549	

As previously mentioned, there was a good prediction of the η_{avg} obtained through a linear fit between the *M* and the *N*. Fig. 3 shows the variation of the *slope a* from Eq. 6. Such shear rate generated a laminar condition as expected for a Newtonian fluid. Effects associated with viscous shear were considered by *slope a*.



Figure 3: Linear relations between average Torque, M (Nm) and rotational speed, N (rpm) over the whole temperature range. The measurements were performed in a Paar Physica UDS200 rheometer.

Slope *a* is included in Table 2 along with, P_{η} (calculated from Eq. 6) and the relative

error. The analogue radius (r = 0.03056 m) was calculated by iterations in Eq. 6 with the function Goal Seek in Excel. The constants K_1 and K_2 obtained were 5902.7 m⁻³ and 0.213 accordingly.

Table 2: Variation of <i>slope a</i> with
temperature range and the inclusion of the
predicted viscosity and the relative error.

T	Slope	Predicted	Ε
(°C)	(Nm/rpm)	Viscosity	
		(Pa.s)	
20	0.000039241	1.088	0.020
30	0.000032465	0.9	0.014
40	0.000027326	0.757	0.002
50	0.000023043	0.639	0.004
60	0.000019978	0.554	0.009

Part 2: Determination of the average viscosity in the fast mixing phase

The Reynolds number (Re) was calculated from Eq. 11 with a density of 1090 kg/m³ (ρ at 20°C for the silicon oil) for some of the *N* values as shown in Table 3. The regime of the fluid was considered fully turbulent⁸ for values of Re above 10 000.

$$Re = \frac{\rho \cdot v \cdot D_{mixer}}{\eta_{avg}} \tag{11}$$

In Eq. 11, v (m/s) is the mean velocity derived from N and D_{mixer} (m) is the diameter of the stirrer (0.044 m) of the JT system.

Table 3: Reynolds number of the silicon oil for several rotational speeds (*N*) in the JT system at 20°C.

Rotational Speed	Reynolds Number	
(rpm)	(Re)	
40	3982	
50	4977	
100	9954	
200	19 909	
300	29 863	

It can be seen from Table 3 that the transition from laminar to turbulent regime occurs between 100 and 200 rpm at 20°C.

In the N range from 100 to 500 rpm, a non-linear trend between M and N was obtained as seen from Fig. 4.



Figure 4: Non-linear relations between average Torque, M (Nm) and rotational speed, N (rpm) over the whole temperature range. The measurements were performed in a Paar Physica UDS200 rheometer.

The torque *M* measured represents the term *z* in Eq. 10. The prediction error *J* was minimized and the constants and the coefficients calculated in MATLAB for the proposed model in Eq. 8, were A = 100.9, B = 0.75, C = 0.032 and D = 2.15. Eq. 12 shows the predicted *M* (or \hat{z} in Eq. 10) with the obtained constants and coefficients. It is hence possible to predict the average viscosity (P_{η}) from Eq. 12 with the model.

$$\hat{z} = 100.9 \cdot N^{0.75} \cdot \eta + 0.032 \cdot N^{2.15} \quad (12)$$

The experimental M (z) versus the predicted $M(\hat{z})$ can be seen in Fig. 5. There is a good prediction of the M and it can be seen from the model (Eq. 12) that there is viscous contribution to the dynamic of the fluid along with a turbulent effect. The latter is reflected in the second term of the Eq. 12 with a coefficient D = 2.15 and the non-linear trend from Fig. 4 can hence be attributed to

the fact that D is positive and higher than 2. Moreover, the second term of Eq. 12 shows that the location of the $\dot{\gamma}_{avg}$ is dependent on N when the turbulent regime is generated.





Although Eq. 12 was obtained from the experimental data in the rapid mixing (100-500 rpm), it expresses the contribution to the M_{avg} from both laminar and turbulent effects at all the speeds (10-500 rpm). There are most likely laminar effects in the layer close to the mixer surface (with fluid velocities close to zero) whereas larger scale turbulent effects may prevail in other sections of the container². Hence, both contributions differ in magnitude over the whole speed range (10-500 rpm).

The P_{η} was calculated from Eq. 12 for all the measured M (25). The E distribution (calculated from Eq. 7) of the P_{η} is shown in Fig. 6.



Figure 6: Relative error distribution of the predicted viscosity for all the experimental measurements for average torque (25) over the whole temperature range (20-60°C) for each rotational speed (100-500 rpm). Measurements 1-5 correspond to 100 rpm, 6-10 to 200 rpm, 11-15 to 300 rpm, 16-20 to 400 rpm and 21-25 to 500 rpm.

It can be seen from Fig. 6 that the *E* has an even variation for *N* between 100 and 200 rpm whereas from 300 rpm onwards, there is a slight increase of it. Therefore, the model is suitable for the prediction of the η_{avg} of a Newtonian fluid in such temperature range in the turbulent regimes from 100 to 500 rpm.

It is suggested as a further step of this research, to test an $\dot{\gamma}_{avg}$ in a flocculation of wastewater in the replicated Jar-Tester system. Secondly, the same $\dot{\gamma}_{avg}$ of this calibration procedure for a water-based fluid (e.g. wastewater) could be replicated in a larger scale (e.g. pilot or industrial) in order to compare the separation efficiency in a flocculation process between the Jar-Tester and a larger scale mixing equipment⁴. Such separation efficiency could be expressed for instance, in terms of changes in M_{avg} in the laminar and turbulent regimes in the flocculation of wastewater.

CONCLUSIONS

A prediction model of the viscosity can be obtained from the replicated JT system for both laminar and turbulent regimes from a standard with known viscosity. However, the calibration procedure of this investigation showed that the location of the average shear rate in turbulent regime was dependent on rotational speed.

The model provides information about the turbulent regime with a component that affects the average shear stress with a nonlinear response from the rotational rheometer.

This investigation is a first step for the understanding of the changes in average viscosity in the laminar and turbulent regime when performing a JT for the flocculation of wastewater.

ACKNOWLEDGMENTS

The authors of this work are thankful with the Norwegian research council as well as with the Norwegian industrial partners, Lyse Neo AS, IVAR IKS, Cambi AS, HØST Verdien i avfall, Lindum AS and Agroplas AS for the funding of this work through the Fixation of Nutrient Elements in Digestate project (grant # ES 459248/0).

REFERENCES

1. Teefy, S., Farmerie, J. and Pyles, E. (2010), "Operational Control of Coagulation and Filtration Processes", 3rd Ed., **2**, pp. 17-18, ISBN 978-1-58321-801-3.

2. Zhang, J., Xu, S. and Li, W. (2012), "High Shear Mixers: A Review of Typical Applications and Studies on Power Draw, Flow Pattern, Energy Dissipation and Transfer Properties", *J. Chem. Eng. and Proc.*, **57-58**, 25-41.

3. Steffe J.F., (1996), "Rheological methods in food process engineering", Freeman Press, East Lansing, Mich., pp. 213-214.

ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY, VOL. 23, 2015

4. Ömerci, B., (2007), "Optimization of a Full-Scale Dewatering Operation Based on the Rheological Characteristics of Wastewater Sludge", *J. Water Research*, **41**, 1243-1252.

5. Salas-Bringas, C., Jeksrud, W.K., Lekang, O.-I. and Schüller, R.B. (2006), "A Calibration Method for a New Type of Rheometer", *Annual Transactions of the Nordic Rheology Society*, **14**, 197-201.

6. Steffe J.F., (1996), "Rheological methods in food process engineering" Freeman Press, East Lansing, Mich., pp. 33-34.

7. Schüller, R.B., Tande, M. and Amundsen, L. (2010), "Experimental determination of time dependent yield properties", *Annual Transactions of the Nordic Rheology Society*, **18**, 25-30.

8. Sinnott, R. K. (2013), "Coulson & Richardson's Chemical Engineering", 4th Ed.,
6, Butterworth-Heinemann, p. 73. ISBN 0-7506-6538-6.