

Article

# On the Use of the Coaxial Cylinders Equivalence for the Measurement of Viscosity in Complex Non-Viscometric, Rotational Geometries

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Received: 19 January 2020; Accepted: 27 March 2020; Published: 1 April 2020



**Abstract:** The rheology of macroscopic particle suspensions is relevant in many industrial applications, such as cement-based suspensions, synthetic and natural drilling fluids. Rheological measurements for these complex, heterogeneous systems are complicated by a double effect of particle size. On the one hand, the smallest characteristic length of the measuring geometry must be larger than the particle size. On the other hand, large particles are prone to sediment, thus calling for the use of rotational tools that are able to keep the suspension as homogeneous as possible. As a consequence, standard viscometric rotational rheometry cannot be used and complex flow geometries are to be implemented. In this way, however, the flow becomes non-viscometric, thus requiring the development of approximate methods to translate the torque vs. rotation speed raw data, which constitute the rheometer output, into viscosity vs. shear rate curves. In this work the Couette analogy methodology is used to establish the above equivalence in the case of two complex, commercial geometries, namely, a double helical ribbon tool and a square-shaped stirrer, which are recommended for the study of relatively large size suspensions. The methodology is based on the concept of the reduction of the complex geometry to an equivalent coaxial cylinder geometry, thus determining a quantitative correspondence between the non-standard situation and the well-known Couette-like conditions. The Couette analogy has been used first to determine the calibration constants of the non-standard geometry by using a Newtonian oil of known viscosity. The constants have been subsequently used to determine the viscosity curves of two non-Newtonian, shear thinning fluids, namely a homogeneous polymer solution and two heterogeneous concentrated suspensions. The results show that the procedure yields a good agreement between the viscosity curves obtained by the reduction method and those measured by a standard viscometric Couette geometry. The calibration constants obtained in this work from the coaxial cylinder analogy are also compared with those provided by the manufacturer, indicating that the calibration can improve the accuracy of the rheometer output.

**Keywords:** rheological measurements; non-viscometric geometries; Couette analogy; shear thinning fluids; suspensions

## 1. Introduction

Fluids used in the construction and Oil and Gas (O&G) industries share many common aspects. Typically, they are suspensions of solid particles in viscous matrices of various nature and are often

characterized by very wide size and shape distributions. Examples include mortars and concrete in the construction field, and drilling fluids in the O&G industry [1,2]. The suspended solid phase determines a complex rheological behavior that, in turn, is not easy to quantify. Due to the broadness of the particle size distribution and to the (often large) density difference between the solid and the suspending liquid, particle settling is a major issue when rheological properties are to be measured [3]. In addition, the large particle size calls for equally large measuring geometries. Therefore, the classical, well-defined rotational rheometer geometries, such as the concentric cylinder geometry, produce wrong results. Therefore, one option for obtaining more accurate shear rate vs. shear stress data is to use unconventional, mixer-type geometries.

A mixer-type rheometer consists of an impeller with a relatively complex geometrical structure, rotating in a fluid contained in a tank, usually a cylindrical cup. The impeller shape and size are designed to minimize sedimentation effects and to allow for measurements on solid suspensions with large characteristic dimensions (typically, larger than one millimeter). This, however, makes the flow in the rheometer complex and non-viscometric. Consequently, both the shear stress and the shear rate, which are necessary to determine the fluid viscosity, are not defined. The instrument only returns a torque and a rotation speed value.

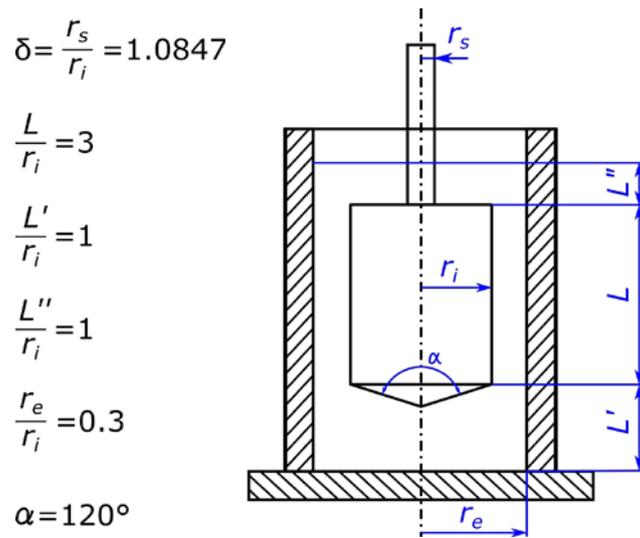
Many examples of mixer-type rheometers can be found in literature [4–9]. In particular, several authors described a procedure to convert the torque and rotational velocity data obtained from a vane-geometry rheometer into shear stress vs. shear rate relationships based on the use of the so-called Couette analogy [2,9–12]. Such a procedure consists in the reduction of the complex impeller geometry to an equivalent concentric cylinder geometry, whose dimensions are chosen in a way to match the viscosity vs. shear rate response of fluids of known rheology.

The main objective of the present work is to apply the above mentioned Couette analogy to the case of two mixer type geometries presently available for a commercial rotational rheometer [13], namely, a double helical ribbon tool and a square-shaped stirrer, indicated by the manufacturer as tools to measure the rheology of large-size concentrated suspensions [14]. The calibration constants for the two geometries are obtained by applying the Couette analogy to a Newtonian fluid and to a non-Newtonian, shear thinning polymer solution, whose rheological responses are quantitatively known. It is shown that the calibration constants provided by the manufacturer are different from those obtained in the present work, and that the latter give more accurate quantitative results for the non-Newtonian fluid. Finally, the two geometries are used to measure the rheology of two drilling fluids suspensions, showing that, when the Couette analogy is used to determine the correct calibration coefficients, both the double helical ribbon geometry and the square-shaped stirrer are able to accurately reproduce the non-Newtonian behavior of the fluids.

## 2. Theoretical Background

In rotational rheometry the so-called Couette, or Coaxial Cylinder (CC), geometry is widely used to determine the viscosity of fluids. Basically, the geometry consists of two coaxial cylinders, one still and the other rotating. Under these conditions, and assuming negligible inertial effects (low Reynolds number) the flow is viscometric, that is, a fluid element is always subjected to the same shear rate. As a consequence, by measuring the torque and the angular velocity at the moving cylinder under steady-state conditions, the viscosity of a Newtonian fluid can be measured [15].

Figure 1 refers to the CC geometry according to the ISO 3219:1995 Standard [16], showing also all the relevant geometrical parameters. In particular, for relatively narrow gaps, both shear rate and shear stress can be considered as uniform across the gap, thus allowing for the determination of the “true” or “representative” viscosity vs. shear rate curve also for non-Newtonian fluids [17]. The Narrow Gap Couette (NGC) geometry is today an international standard for the measurement of the non-Newtonian viscosity of polymers, emulsions and dispersions.



**Figure 1.** The Concentric Cylinder (Couette) geometry and the geometry parameters according to the ISO 3219 Standard.

The NGC rheometry is governed by the following equations:

$$\sigma = \frac{1 + \delta^2}{\delta^2} \frac{M}{4\pi L r_i^2 C_L} \tag{1}$$

$$\dot{\gamma} = \frac{1 + \delta^2}{\delta^2 - 1} \omega \tag{2}$$

where  $\sigma$  and  $\dot{\gamma}$  are the shear stress and the shear rate, respectively,  $\delta = r_e/r_i$  is the ratio between the outer and inner radius,  $M$  and  $\omega$  the measured torque and rotation speed, and  $L$  the height of the inner cylinder.  $C_L$  is an end-effect correction factor accounting for the torque acting at the end faces of the measuring system and is typically taken to be  $C_L = 1.1$  [16].

All geometrical factors appearing in Equations (1) and (2) can be grouped into two constants,  $c_{SS}$  and  $c_{SR}$ :

$$c_{SS} = \frac{1 + \delta^2}{\delta^2} \frac{1}{4\pi L r_i^2 C_L} \tag{3}$$

$$c_{SR} = \frac{1 + \delta^2}{\delta^2 - 1} \tag{4}$$

Finally, the fluid viscosity,  $\eta$ , can be obtained as:

$$c_{SS} = \frac{\delta^2 - 1}{\delta^2} \frac{1}{4\pi L r_i^2 C_L} = \frac{c_{SS}}{c_{SR}} \frac{M}{\omega} = K \frac{M}{\omega} \tag{5}$$

For a Newtonian fluid, for which the ratio between torque and rotation rate is a constant,  $K$  is the only geometrical constant to be known to determine the viscosity. In the non-Newtonian case, on the contrary, knowledge of  $c_{SS}$  and  $c_{SR}$  is required.

The well-defined situation of the NGC geometry finds no correspondence in the case of complex rotational geometries. Here, the flow field is a complex, non-uniform combination of shear and extensional components. The specific shape and size of the rotor, coupled to non-Newtonian characters of non-Newtonian fluid such as suspensions, make it impossible to determine the viscosity from simple formulas like Equation (5). In these cases, only numerical simulation techniques can in principle allow for a quantitative description of the flow characteristics. If, however, an approximate solution to the problem is sought, the so-called “Couette analogy” approach can be followed [9,11]. The approach

is based on the simple idea that the complex geometry can be *reduced* to a virtual coaxial cylinder geometry such that, for a given rotation rate, the complex geometry and its CC virtual equivalent produce the same torque.

In its simplest form [9], the Couette analogy assumes that the fluid is Newtonian and that the geometry consists of an outer cylindrical cup, of radius  $r_e$ , where the rotor is immersed in the fluid up to a given height  $L$ . The complexity of the geometry resides in the inner rotating tool of non-cylindrical shape. Let now  $\omega$  and  $M$  be the rotation rate and the torque measured by the complex geometry on a fluid of known viscosity,  $\eta$ . According to the Couette analogy, it is assumed that the non-viscometric flow is equivalent to that taking place in a virtual coaxial cylinder geometry with the same  $r_e$  and  $L$  and of unknown inner radius  $r_{i,eq}$ . The latter can be found by rewriting Equation (5) as:

$$4\pi LC_L \eta_{eq} \frac{\omega}{M} = \frac{r_e^2 - r_{i,eq}^2}{r_e^2 r_{eq}^2} \quad (6)$$

so that the inner radius of the equivalent Couette geometry is given by:

$$r_{i,eq} = \frac{r_e}{\sqrt{1 + 4\pi LC_L \eta_{eq} r_e^2 \frac{\omega}{M}}} = \frac{r_e}{\sqrt{1 + 4\pi LC_L \eta_{eq} K r_e^2}} \quad (7)$$

In the second equality of Equation (7) use has been made of the definition of the calibration constant  $K$ . This proves that, in order to determine the Couette analogue of the complex geometry for the case of a Newtonian fluid, only the calibration constant of the non-viscometric geometry is required.

The reduction of a complex rotational geometry to its Couette equivalent holds in principle only for the case of a Newtonian fluid. Attempts have been made in the literature to include the non-Newtonian constitutive behavior into the analogy [9,12]. However, adding the non-Newtonian complexity to a model that is already based on very crude assumptions is not particularly meaningful. For this reason, in order to validate the model for the complex geometries used here, in the next section the experimental results for both Newtonian and non-Newtonian fluids are compared by keeping the Couette analogy at its simplest, yet more affordable and robust level described above.

### 3. Materials and Methods

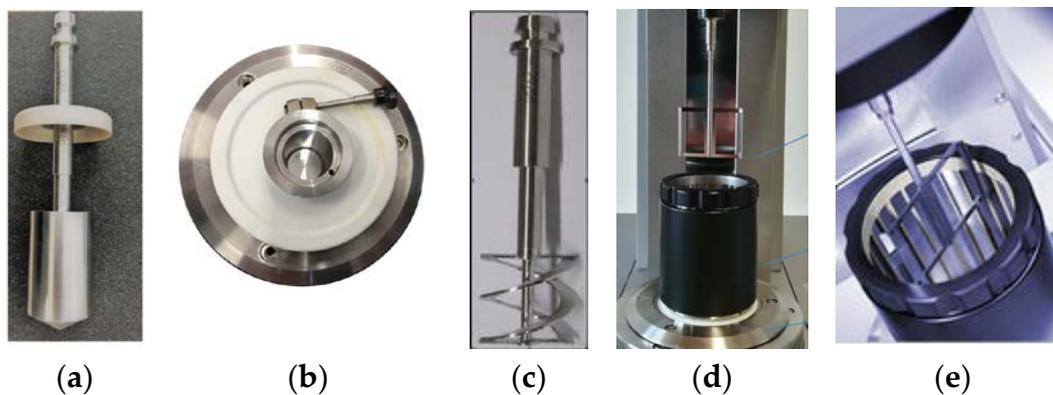
Four different fluids were used to test the non-viscometric geometries. They are:

- (1) A Newtonian silicon oil (BDH1000, from Merck KGaA, Darmstadt, Germany) with a viscosity of about 1 Pa s (labeled SO);
- (2) A shear thinning, homogeneous aqueous solution of HydroxyEthyl Methyl Cellulose (TyloseMH600046P6, manufactured by SE Tylose GmbH, Wiesbaden, Germany). The concentration is 0.7% wt, which determines (see data of Figure 7) a Newtonian plateau viscosity of about 0.7 Pa-s (labeled HEMC);
- (3) Two commercial drilling fluids provided by Geolog Srl (Milan, Italy). They are a water-based (labeled WBM) and an oil-based (OBM) suspension containing sand particles of variable size up to about 1 mm and a volume fraction of about 10%. The suspending fluid contains several additives, including polymers, but their detailed formulation is not known for confidential reasons. Both suspensions show a strong shear thinning behavior over a wide range of shear rates and no hint of a low shear Newtonian plateau.

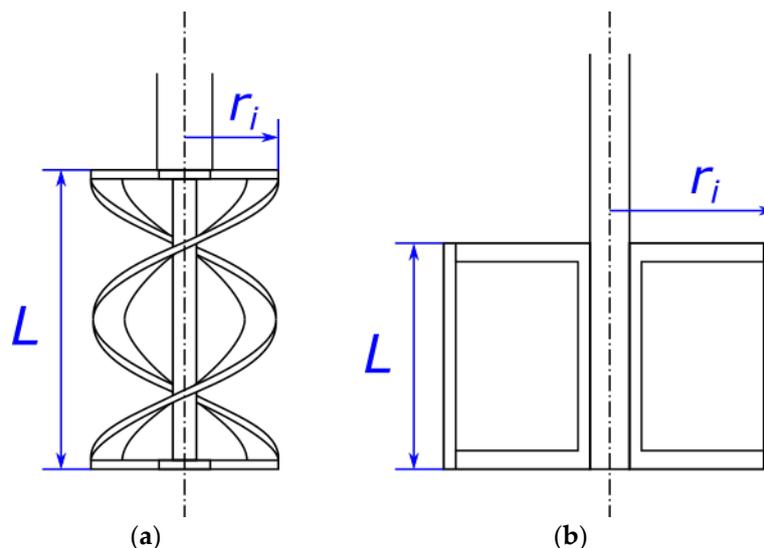
All fluids were tested by an Anton Paar MCR702 rotational rheometer (Anton Paar GmbH, Graz, Austria). The rheometer is equipped with Peltier units for an accurate thermal control. In all experiments, temperature has been kept constant at 25 °C. Measurements were repeated at least four times for each fluid and each geometry. In all cases, a very good reproducibility was obtained. For this reason, only single run experiments are shown.

Three different rotational geometries have been used, manufactured by Anton Paar for the MCR rheometer line. Pictures and drawings of the rotors are given in Figures 2 and 3. They are:

- (1) A Narrow Gap Couette geometry (NGC) formed by an inner cylinder (Figure 2a) and an outer cup (Figure 2b). The geometry conforms to the ISO 3219 Standard and has been used to calibrate the other geometries via the Couette analogy;
- (2) A Double Helix (DX) rotor (Figure 2c) that uses the same cup of the NGC. The shape of the DH rotor introduces a continuous flow from the bottom to the top of the cell, thus opposing particle sedimentation;
- (3) A flat, square-shaped blade rotor (Building Material Cell, BMC, Figure 2c–e) fitted in a cylindrical cup larger than that used by the NGC and DH configurations. The cup includes an inner cage to prevent slippage of the fluid. It must be noticed that, according to the manufacturer, the tool can be used to measure suspensions having aggregates no larger than 5 mm. Other rotors are available for larger particle suspensions.



**Figure 2.** The rotors used in this work: (a) Narrow Gap Couette (ISO 3219); (b) Top view of the cup used for both the Narrow Gap Couette (NGC) and the Double Helix geometry; (c) Double Helix (DH); (d) Building Material Cell (BMC); (e) A close-up of the BMC cup.



**Figure 3.** Drawing of the two complex rotors and their main dimensions: (a) Double Helix (DH); (b) Building Material Cell (BMC).

Drawings of the DH and BMC rotors are shown in Figure 3 along with their main geometrical dimensions (the same drawing for the NGC has been already reported in the previous Figure 1). Dimensions of inner and outer radii and rotors height are summarized in Table 1.

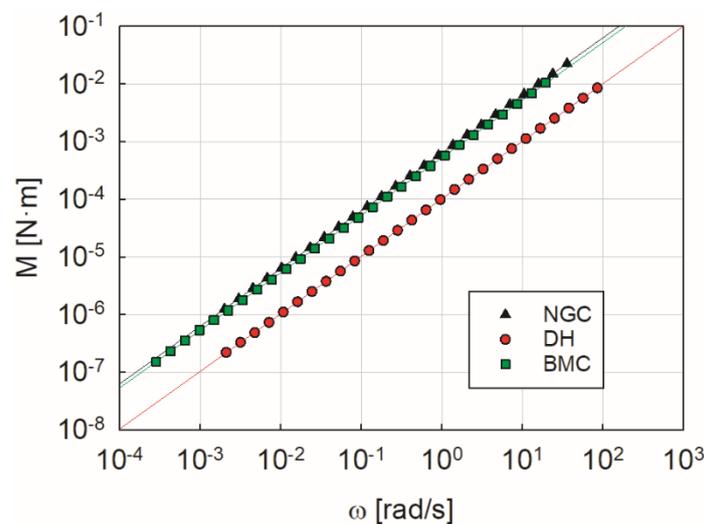
**Table 1.** Main geometrical parameters of the three geometries. The equivalent internal radius, obtained from the Couette analogy procedure described in the text, is also reported for the two non-conventional geometries.

Geometry	$r_e$ (mm)	$r_i$ (mm)	$r_{i,eq}$ (mm)	$L$ (mm)
NGC	14.46	13.33	/	39.98
DH	14.46	12.00	10.446	37.00
BMC	35.0	29.5	23.358	44.3

#### 4. Experimental Results and Discussion

##### 4.1. Calibration of Non-Conventional Geometries

The Newtonian silicon oil has been used to calibrate the complex geometries and to determine the corresponding coefficients. To this end, the NGC geometry has been used as a reference. Figure 4 shows the steady-state torque as a function of the rotation speed for the three rotors.



**Figure 4.** Torque as a function of rotation rate for the three geometries. The straight solid lines are linear regressions of the data.

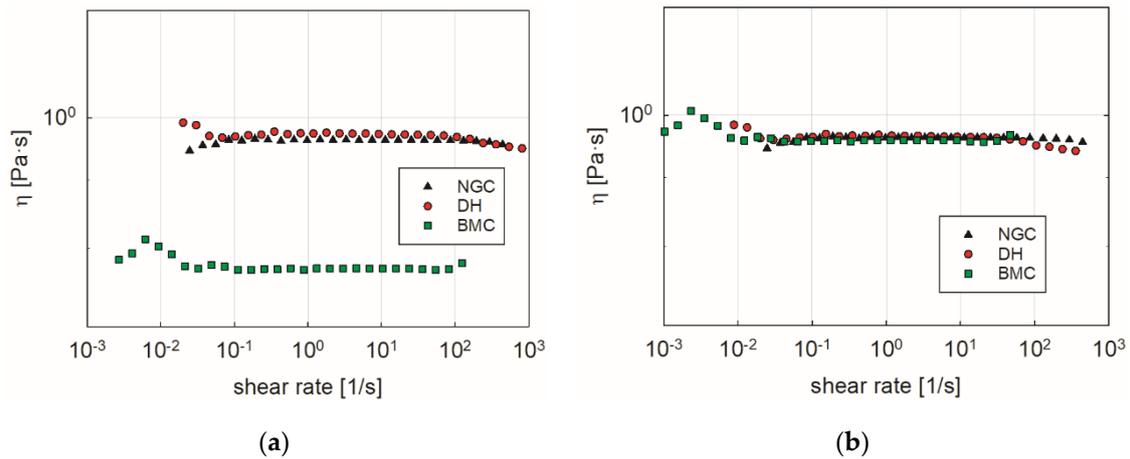
For all three geometries the log-log data are well described by straight regression lines of slopes equal to one (within three significant digits) over about five decades of rotation rate and torque, indicating an almost perfect linearity. This is an expected result, in light of the Newtonian character of the SO fluid.

The data in Figure 4 can now be converted into viscosity vs. shear rate curves. To do that, Anton Paar provides the constants  $c_{SS}$  and  $c_{SR}$  for the three geometries. They are listed in Table 2. While the constants for the NGC geometry are rigorously determined from Equations (3) and (4), those provided for the DH and BMC rotors are empirical, although no details of the calibration procedure are given.

**Table 2.** The geometrical constants  $c_{SS}$  and  $c_{SR}$  for the three geometries. The values of  $c_{SS}$  and  $c_{SR}$  provided by Anton Paar are also reported along with those obtained in this work. The calibration constants  $K$  for the DH and the BMC derived from the calibration procedure are also reported.

Geometry	$c_{SS}$ (1/m <sup>3</sup> )		$c_{SR}$ (1/rad)		$K$ (rad/m <sup>3</sup> )
	Anton Paar	Couette Analogy	Anton Paar	Couette Analogy	
-	-	-	-	-	-
NGC	18,847	-	12.337	-	-
DH	90,700	39,420	9.5493	4.1834	9422.2
BMC	14,070	6584.8	9.5493	3.6061	1826.0

Figure 5a shows the viscosity of the silicon oil as a function of shear rate for the NGC and the two complex geometries, obtained by using the standard Anton Paar geometrical coefficients. It is apparent that the three geometries return different values of the viscosity. The viscosity measured by the NGC tool corresponds to the value provided by the oil manufacturer. In particular, the NGC geometry returns an average viscosity  $\eta = 0.9604$  Pa·s. The viscosity obtained from the DH and the BMC substantially differ from each other. In particular, the viscosity of the DH system matches the true value obtained by the NGC geometry, whereas the BMC system underestimates the viscosity by about 20%. This means that, at least for the BMC rotor, the coefficients  $c_{SS}$  and  $c_{SR}$  provided by the rheometer manufacturer are not accurate.



**Figure 5.** The viscosity of the silicon oil (SO) fluid as a function of shear rate for the NGC and the two complex geometries. (a) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients provided by Anton Paar; (b) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients from the Couette analogy.

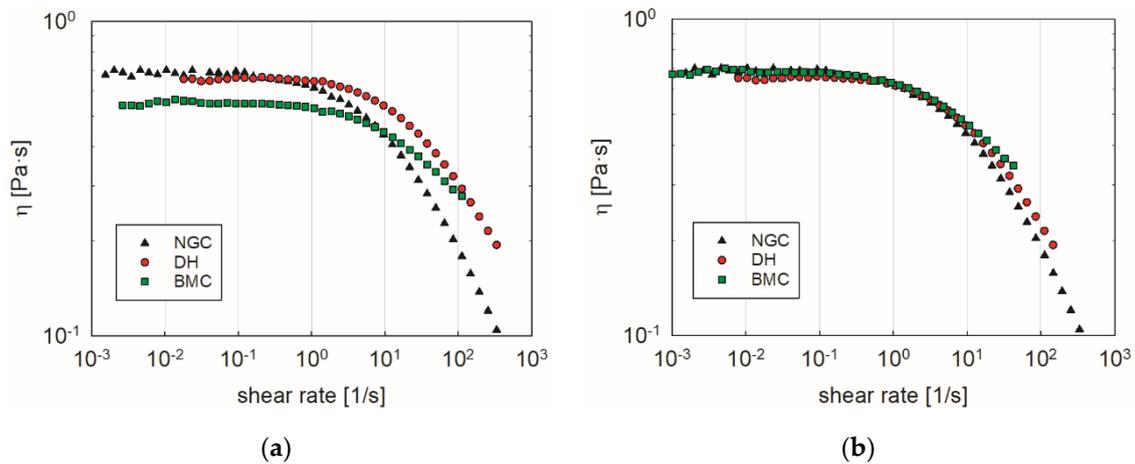
Having verified that the NGC geometry returns the true viscosity value, the latter has been used to determine the new  $c_{SS}$  and  $c_{SR}$  coefficients for the DH and BMC tools based on the Couette analogy procedure described in Section 2. First, for each geometry the calibration constant  $K$  has been calculated from equation (5). To this end, the average viscosity measured with the NGC geometry is used, along with the slope of the torque vs. rotation rate linear plot (see Figure 4). Then, the Couette analogy is applied and the inner radius of the virtual concentric cylinder analogue,  $r_{i,eq}$ , is calculated by using Equation (7), where the necessary geometrical parameters are those listed in Table 1. Finally, use of Equations (3) and (4) allows for the determination of the two coefficients  $c_{SS}$  and  $c_{SR}$ .

The numerical values resulting from the above procedure are listed in Table 1 ( $r_{i,eq}$ ) and Table 2 ( $K$ ,  $c_{SS}$  and  $c_{SR}$ ). It can be noticed that the coefficients calculated by using the Couette analogy are substantially different from those provided by Anton Paar. Figure 5b shows the viscosity curves obtained from the Couette analogy. All experimental data, including those obtained with the BMC geometry, now superimpose on those measured by the NGC geometry. This is an obvious result, as the analogy is based on the assumption that the correct viscosity value is the one measured by the NGC geometry, which is then used to "constrain" the coefficients of the other geometries to return the correct viscosity value. Notice, however, that the experimental data for the two non-viscometric geometries are shifted along the shear rate axis, due to the change in the  $c_{SR}$  coefficient.

#### 4.2. Validation of the Couette Analogy Calibration Procedure

Once the  $c_{SS}$  and  $c_{SR}$  coefficients for each geometry are determined by the Couette analogy in a way to predict the correct Newtonian viscosity, they can be used to compare the NGC measurements performed on non-Newtonian fluids with those of the non-viscometric, complex rotors.

The results of the viscosity measurements for the shear thinning, HEMC solutions are reported in Figure 6. As in the previous Figure 5, the viscosity vs. shear rate curves obtained by using the geometry coefficients provided by Anton Paar are compared with those calculated from the Couette analogy.

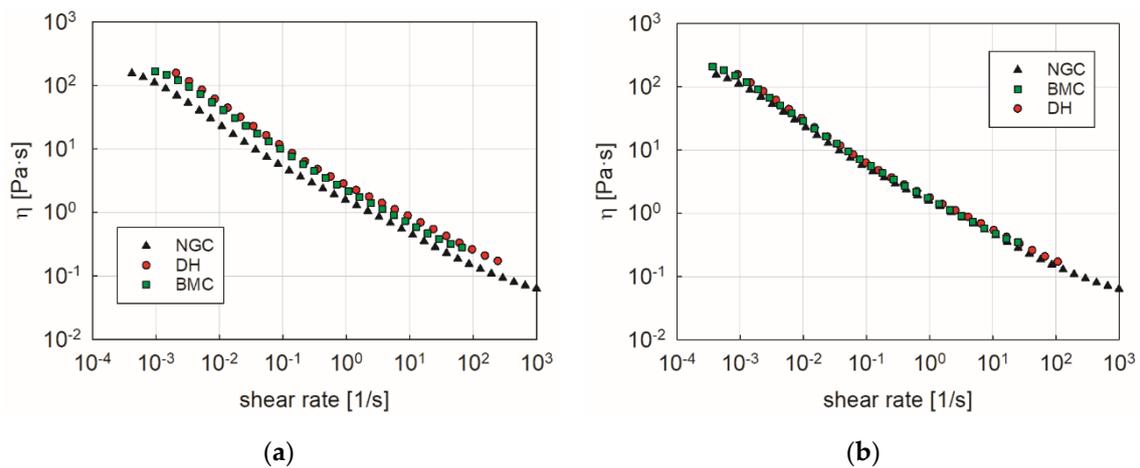


**Figure 6.** The viscosity of the HydroxyEthyl Methyl Cellulose (HEMC) aqueous solution as a function of shear rate for the NGC and the two complex geometries. (a) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients provided by Anton Paar; (b) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients from the Couette analogy.

When the Anton Paar constants are used (Figure 6a), substantial discrepancies are found between the NGC results and those obtained with the complex rotational geometries. In particular, the BMC impeller shows a Newtonian plateau viscosity lower than that measured with the NGC geometry, with a behavior similar to the one already observed for the silicon oil. The relative error is also in this case of the order of 20%. At higher shear rates, on the contrary, the viscosity from the BMC geometry becomes larger than that measured with the NGC, a cross-over between the two curves taking place at a shear rate of ca.  $1 \text{ s}^{-1}$ . As far as the DH geometry is concerned, the Newtonian plateau viscosity is well predicted as for the silicon oil measurements, but the shear thinning part of the curve is shifted to higher shear rates with respect to the reference NGC geometry data.

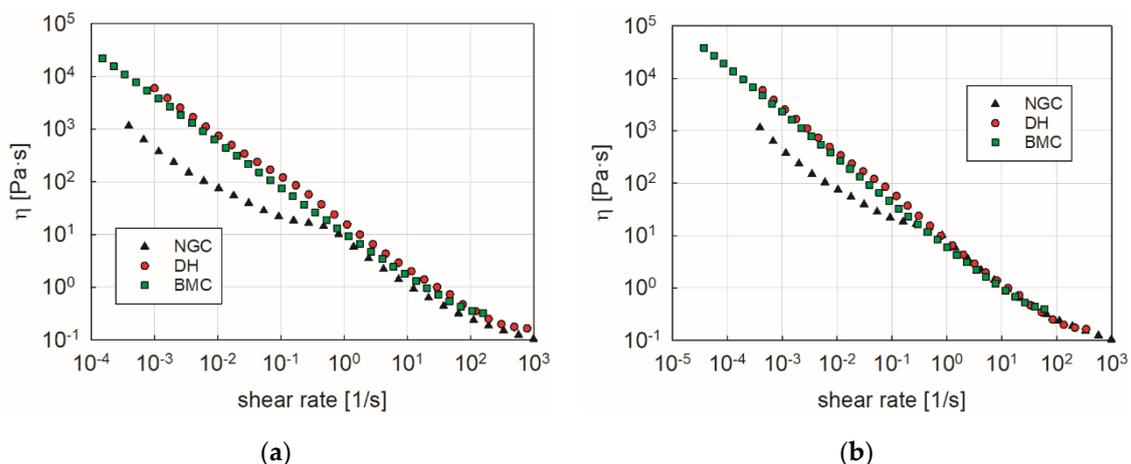
The results improve considerably when the  $c_{SS}$  and  $c_{SR}$  coefficients coming from the Couette analogy are used, as shown in Figure 6b. In this case, the viscosity curves obtained with the DH and BMC rotors match very closely the true viscosity data obtained by the NGC geometry. The agreement is particularly good for the Double Helix, whereas the deviation is larger for the BMC impeller. The DH data clearly indicate that the main reason for measurement improvement derives mostly from the horizontal shift in the data when the Couette analogy is used. This proves that the analogy allows for a robust estimate of the characteristic velocity gradient taking place in these non-viscometric, complex flow geometries.

The data presented in Figure 6 confirm that, even for a shear thinning homogeneous fluid, the Couette analogy provides an excellent interpretation of the experimental data. The situation becomes more challenging in the case of the non-Newtonian drilling fluid suspensions. Figure 7 reports the viscosity as a function of shear rate for the WBM suspension. In this case, where a non-Newtonian plateau is not present, at least in the shear rate range explored, both the DH and BMC viscosity qualitatively follow the NGC geometry behavior when the factory coefficients are used (Figure 7a). The agreement becomes remarkable when the values of  $c_{SS}$  and  $c_{SR}$  are calculated by the Couette analogy procedure (Figure 7b).



**Figure 7.** The viscosity of the WBM drilling fluid suspension as a function of shear rate for the NGC and the two complex geometries. (a) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients provided by Anton Paar; (b) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients from the Couette analogy.

Figure 8 shows the last set of viscosity measurements, involving the OBM suspension. In this case the experimental results are more contradictory. In the high shear rate region, above about  $1 \text{ s}^{-1}$ , the agreement between the three geometries is qualitatively good when the Anton Paar coefficients are used and becomes quantitatively excellent when the Couette analogy is implemented. On the other hand, when the low shear rate region is considered, the NGC data strongly differ from those obtained from the DH and BMC geometries. The discrepancy, in this case, is probably due to an incorrect evaluation of the viscosity in the NGC tool. One possible explanation is that, due to the non-polar character of the suspending fluid (oil) with respect to the electrically active sand particles, the system is prone to formation of particle aggregates at low rotation rates. Such aggregates are excluded from the relatively narrow gap measuring region of the concentric cylinders, thus determining an apparent lower viscosity of the suspension. Conversely, at higher shear rates the agglomerates are probably destroyed by the action of flow, thus restoring the well-dispersed suspension condition and, as a consequence, the correct viscosity behavior.



**Figure 8.** The viscosity of the OBM drilling fluid suspension as a function of shear rate for the NGC and the two complex geometries. (a) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients provided by Anton Paar; (b) Results obtained by using the  $c_{SS}$  and  $c_{SR}$  coefficients from the Couette analogy.

### 5. Conclusions

The main conclusion of this work is that the Couette analogy concept, which allows for the reduction of a complex, non-viscometric rotational geometry to a virtual concentric cylinder analogue,

can be successfully applied to determine the viscosity vs. shear rate curve of non-Newtonian fluids in complex geometries. The methodology has been applied to two commercial rheological tools, namely, a Double Helix (DH) and a flat square-shaped (BMC) rotor. It has been found that the standard geometrical coefficients provided by Anton Paar to convert the torque vs. rotation rate measurements into viscosity vs. shear rate curves do not produce accurate predictions. On the contrary, once the Couette analogy is implemented by using a Newtonian fluid as a rheological reference, the two complex geometries are able to reproduce, to a great level of accuracy, the non-Newtonian behavior of homogeneous as well as heterogeneous, suspension-based fluids. It must be also remarked that, although the non-conventional geometries are characterized by a complex flow field, including both shear and extensional non uniform components, the Couette analogy procedure allows for an accurate description of the viscous behavior of complex fluids over a very wide range of shear rates, sometimes extending over as many as eight decades.

**Author Contributions:** Conceptualization and methodology, N.G.; formal analysis, N.G. and J.L.; investigation, R.M.P., S.C. and V.V.; resources, E.R.R.; data curation, S.C. and N.G.; writing—original draft preparation, R.M.P.; writing—review and editing, N.G.; supervision, E.R.R. and N.G. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Acknowledgments:** We are grateful to Michela Brunelli for performing some of the rheological measurements and to Anton Paar GmbH for providing all technical specifications of the geometries tested in this work.

**Conflicts of Interest:** R.M.P. and E.R.R. are employees of Geolog. J.L. is an employee of Anton Paar. All of them state that in this paper there is nothing that may be considered as a conflict of interest.

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