1 Introduction

Since the breakthrough of Bingham in the late 20's of the 20^{th} century when he modeled for the first time a *non–Newtonian fluid*, rheology , a branch of continuum mechanics, has been evolving in the study of the deformation of matter. This study covers not only the measuring of the deformation of matter under a certain applied stress or strain but also its use, modeling and understanding.

In order to characterize a material, data need to be collected from measurements. These data should allow to reproduce the deformation of the material and understand how this deformation occurs. This step encloses the modeling of the material property of interest, e.g. the viscosity. One correlates forces acting on the fluid (e.g. torque) with the deformation rate (shear rate) and the resulting relationship between these two variables provides information concerning the viscosity.

To describe the behavior of a material and collect data experimentally a rheometer is necessary. In this device one tries to create ideal flow conditions of the fluid. An example is provided by a rotational rheometer.
Rotational rheometers are well established devices which have been widely used in research as well as in industry.

As mentioned above, for the case of the viscosity one tries to establish a function of the form

$$\eta = f(\frac{M}{\Omega}) \,, \tag{1.1}$$

where M stands for the torque applied and Ω for the angular velocity measured during the test.

In rotational rheometers it is sometimes necessary to carry out the tests up to relative high velocities or torques. This implies that secondary motions have to be reckoned with. In rheometers such as the (one–gap) concentric cylinder (bob–in–cup geometry) this type of flow will deliver wrong values for the viscosity of the fluid. Although this is a limiting factor this is not the only problem when measuring the viscosity of a fluid with a bob–in–cup system. The so–called wall slip may also occur at the surface of the cylinders.

One can say that slip occurs when a thin layer or film forms on the shearing surface so that the typical equations used to estimate the viscosity with the bob–in–cup system, based on the assumption of non–slip at the walls will not allow to predict the viscosity. It is possible to overcome these obstacles just by limiting the measuring range or modifying the surface of the cylinders. Of course this is not always the best and simplest way to overcome these issues. Often one encounters suspensions where special care has to be paid to avoid disrupting the sample during the immersion of the bob.

An alternative to the bob–in–cup geometry is the vane–in–cup geometry. In this geometry any slip effect at the vane can be excluded and less breakage of the structures –in the case of suspensions– will occur upon immersion of the rotating body. The sample is kept homogeneous. In the present work we investigate a 4–bladed vane immersed in a cup. Despite its simplicity in construction its use has been restricted until now almost only to measuring the yield stress .

Since the vane–flow is not viscometric there is no simple analytic solution for estimating the shear rate if one wants to calculate the deformation as done with the bob–in–cup geometry. Any application of the equations that are employed for rotational rheometers to estimate the shear rate is bound to lead to wrong values.

One way of calculating the shear rate is via numerical simulations which also serve to validate experimental data and / or rheological models. Computational fluid dynamics (CFD) has been developing continuously during the last twenty years with an increasing tendency toward non–Newtonian rheology. Nowadays powerful numerical techniques and hardware are available to investigate relatively complex flows. This allows us to overcome experimental limitations encountered in rheometry due to restrictions imposed by the equipment or measuring techniques.

In the present work experimental and numerical investigations are carried out for vane flow. In this context special attention is paid to its use for material characterization. The whole work is structured as follows.

Chapter 2 gives an overview of the work performed with the vane geometry in the last twenty years. There, a summary is given of the direction investigations have taken to measure the yield stress and attempts to measure the viscosity of fluids.

Chapter 3 is devoted to the numerical aspects to perform the simulations for vane flow. The finite volume method (FVM) and the discretization approach to solve the governing equations are presented. The non–dimensional scaling of the general transport equation, which is the basis for our simulations, is also derived and at the end of the chapter, the software used for the simulations, the pre and post–processing is also briefly described.

Chapter 4 introduces the vane–in–cup geometry. Here are imposed the boundary conditions of the problem, the mesh of the geometry and some theoretical aspects for its general use in rheological material–characterization.

In chapter 5 the methodology of the optical measuring technique used for the visualization of the flow is described. Here we are concerned with the elementary procedure for image acquisition, statistical validation and analysis of data for the estimation of the displacement of the tracer particles present in the fluid.

Chapter 6 is concerned with Newtonian and non–Newtonian fluids considering their chemical and physical properties. The reference viscometer, namely the bob–in–cup system which was used to characterize the different fluids is shown schematically under the usual assumptions to calculate the shear stress and the viscosity of a fluid and compared to the vane–in–cup geometry. In addition, the experimental set–up including the vane viscometer is schematically represented.

Chapter 7 deals with the rheological material–characterization of the Newtonian and non–Newtonian fluids. In chapter 8 our approach in estimating the device constants for the vane–in–cup geometry for its use as viscometer is explained and demonstrated. The flow curves of non–Newtonian fluids modeled in chapter 7 are compared with those computed with our approach and the usage of the vane as viscometer is shown.

Finally in chapters 9 and 10 the results of the experimental visualization and numerical simulations for the vane flow at certain regions of the flow domain are presented and discussed.

2 Literature survey

In the last twenty years many authors attempted to develop methods to use the vane for material characterization in a rheological context. These methods were developed or implemented to measure the yield stress or the viscosity of fluids. A consequence of these methods is a self–imposed dependence upon the rheological model used to calculate the device constants. All these works can be divided into three main categories: experimental, theoretical and numerical.

Experimental investigations have been devoted to characterize materials and to determine the device constants. The efforts of theoretical and numerical studies try to describe the flow behavior in the vane–in–cup system considering material properties and / or the geometry. Theoretical studies show that the mathematical treatment of the vane is complex. Whatever the investigations were dealing with there were always common assumptions about the type of flow.

The following section gives an overview of the most important research work carried out for the vane flow in the last twenty years. Table 2.1 summarizes these works.

Table 2.1: Overview of the research–work carried out with the vane geometry since 1983.

Author	**Year**	**Material**	**Approach**
Nguyen et al. [58]	1983	Bingham, Casson, Herschel–Bulkley, Buckingham–Reiner	Experimental (Yield stress)
Keentok et al. [48]	1985	Bingham	Numerical (Shear surface)
Nguyen et al. [59]	1985	Bingham, Casson, Herschel–Bulkley	Experimental (Yield stress)

Continued on next page

Author	Year	Material	Approach
Yoshimura et al. [83]	1987	Suspensions	Experimental (Yield stress)
Haimoni et al. [39]	1988	Cement slurry	Experimental (Yield stress)
Barnes et al. [11]	1990	Newtonian, Ostwald de Waele	Numerical
Alderman et al. [2]	1991	Clay suspensions	Experimental (Yield stress)
Sherwood et al. [72]	1991	Colloidal suspensions, Newtonian	Theoretical
Castell–Perez et al. [23]	1990	Newtonian, Ostwald de Waele	Experimental (Device constants)
Atkinson et al. [5]	1992	Newtonian, linear elastic	Theoretical (Stress, Flow)
Briggs et al. [18]	1996	Frozen ice	Experimental (Yield stress)
Liddell et al. [62]	1996	Suspensions	Experimental (Yield stress)
Yan et al. [80]	1997	Herschel–Bulkley, Casson, Maxwell	Numerical (Yield surface)
Daubert et al. [28]	1998	Food products	Numerical (Yield stress)
Perez et al. [63]	1999	Red mud, soft clay	Numerical (Stresses)
Glenn III et al. [36]	2000	Newtonian,	Experimental

Continued on next page

Author	Year	Material	Approach
		Ostwald de Waele	(Device constants)
Bravian et al. [15]	2002	Newonian, Power–law	Experimental (Yield stress, Device constants)
Rolon–Garrido et al. [69]	2002	Micellar solutions	Experimental (Flow stability)
Farias et al. [33]	2004	Polymer solutions	Experimental (Material characterization)
Krulis et al. [49]	2004	Flavored yoghurt, Newtonian	Experimental (Device constants)
Martinez–Padilla et al. [53]	2004	Sauces	Experimental (Material characterization)

Nguyen et al. ([58], [59]) introduced at the beginning of the eighties a relatively new method for measuring the yield stress with the vane–in–cup geometry. This method consisted in treating the rotating vane as an imaginary cylinder [1]. This facilitated the calculation of the torque exerted on the vane since the equations used to calculate stress distributions in a cylinder were directly applied. In this experimental work they performed a series of measurements with high liquid–solid suspensions whose flow behavior could be predicted with different constitutive equations namely the Bingham, Herschel–Bulkley, Buckingham–Reiner and Casson models. They also considered a 'correction' since the vane diameter in practice does not approach zero, that is, they took into account a non–uniform stress–distribution effect over the ends of the cylinder.

Following the assumptions introduced by Nguyen et al., Keentok ([48]) carried out

[1] This has been an usual assumption in soil mechanics ([59]).

numerical simulations with a finite–element (FE) code on a four–bladed vane as well as some experimental work. He concentrated his work on the fracture zone (or surface of shearing) around the four–bladed rotating vane in a Bingham fluid. According to his numerical simulations the region where shearing occurs has a diameter slightly larger than the vane diameter and was about 2.5% larger than the vane diameter. No information is given about the mesh employed. From the results of his experiments he claims that the diameter of the shearing surface was 1.00–1.05 times larger than the vane diameter. He employed for his experiments automotive greases (represented by a Bingham model). Furthermore, he reports he was able to photograph the flow in the vane rheometer and that the fractural surface was approximately cylindrical, but this is not clear how it was conducted.

Although he affirms that he got good agreement between his experimental and numerical works he did not find any theoretical relationship between theory and experiment. For example, the fact that the shearing–surface diameter increased in experiments linearly with the ratio yield–stress to plastic viscosity but numerical simulations showed the contrary, an exponential decrease with increasing ratio yield–stress to plastic viscosity. Despite these discrepancies he recommended the use of the vane for yield stress measurements if a diameter correction is applied.

Yoshimura et al. ([83]) compared the vane technique with other standard methods to measure the yield stress. According to them, values obtained for different oil-in-water emulsions with the vane geometry were of high precision. They pointed out the difficulty in obtaining viscosity information of the fluid after the material starts to flow since the flow field around the vane is quite complex.

Haimoni et al. ([39]) performed yield–stress measurements with a six–bladed vane on a cement slurry. It is well known that cement slurries change their behavior from liquid to solid in matter of hours. Measurements were performed long before reaching the solid state but the samples were given enough time to rest, so that the structures could strengthen. They discuss the shear stress distribution on the vane edges. Different theoretical models to describe this distribution are described: uniform (square), triangular (zero at the vane center having its maximum at the vane tip), parabolic or exponential.

In order to have an uniform stress distribution a large ratio height–to–diameter seems to be required. On the other hand if this ratio is small (less than 2.0) the stress distribution should deviate strongly from a square–uniform distribution and tends to become triangular, depending on the type of material. In practice these distributions will deviate along the vertical and the horizontal edges of the vane, depending on the type of material. They compared all measurements with a bob–in–cup rheometer and attributed the differences between both to slip at the bob.

Barnes [11] carried out simulations with a FE code for a four–bladed vane and a power–law fluid (shear thinning, index $n \leqslant 0.5$). He compared his results with those obtained with a bob–in–cup geometry. The shear stress calculated at the cup wall was equal for both geometries for a given rotational speed. He obtained equivalent flow curves at low shear rates with the same fluid but a sudden viscosity drop was found with the bob geometry at low shear rates. This affect was atributed to wall slip. Since this effect was not observed with the vane he noted this geometry as a valuable tool when slip occurs.

Alderman et al. ([2]) performed experimental investigations to measure the yield stress of aqueous bentonite clay suspensions. They found that there is no end contribution from the blades for the lower yield stress muds something that is different with the findings by Nguyen et al. ([59]). A comparison with a Carrimed bob–in–cup rheometer suggests that the vane technique may be suitable only for yield stresses greater than a minimum value, most likely dependent on the number of blades. The accuracy of the measurements was not only attributed to the number of blades (a four bladed–vane was used) but also to the concentration of the suspensions. The failure of the measurement for lower concentrations was attributed to the simplified assumptions in the vane technique. For high concentrations the divergence in the measurements between the vane–in–cup and the bob–in–cup geometries was attributed to slip at the walls of the last system.

The results of Alderman et al. ([2]) provided an important basis for Sherwood et al. ([72]). Sherwood et al. showed that the vane could not only be used to measure the yield stress of concentrated suspensions but also to obtain the shear modulus of the suspension (treated as a linear–elastic solid) in small deformations. Sherwood et al. studied different N–bladed [2] configurations of the vane imposing slip and non–slip conditions for each different case. With the use of a complex variable analysis they came out with certain simplified results for 2,3,4, and 6–bladed vanes. Their results show that despite the simplicity of the geometry of the vane the mathematical analysis of the vane is not trivial and for practical purposes assumptions and / or simplifications are necessary.

Castell–Perez et al. ([23]) performed a series of measurements with 10 different vane geometries. The height of the vane was kept constant while the ratio vane–diameter to cup–diameter was varied. Their results show that shear rates evaluated with an averaged constant were both, geometry and material dependent. Their results are limited to Newtonian and Oswald–de–Waele fluids.

[2] N denotes the number of vanes.

Atkinson et al. ([5]) went further with theoretical analysis. Using Mellin transforms and the Wiener–Hopf technique they investigated different N-bladed configurations and different boundary conditions. Their interest was the stress distribution and the flow for a 2–D vane.

Briggs et al. ([18]) give an example of the use of the vane in food technology. They consider the vane technique to be advantageous for testing the characteristics of frozen ice cream, mainly for the reason that the vane does not destroy the product structure when the vane is immersed in the sample. Neglecting any end–effects as proposed by Nguyen et al. ([58], [59]), they assumed a solid–body rotation to measure the yield stress of the frozen ice.

Liddell et al. ([62]) performed yield–stress measurements of T_iO_2 suspensions with the vane geometry under the same solid–body rotation assumption. They pointed out that this assumption is "slightly incorrect". Although Nguyen et al. ([59]) have already addressed a non–uniform stress distribution at the ends of the imaginary circumscribed cylinder and Alderman et al. ([2]) minimize its influence on the measuring of the yield stress. Results of Liddell et al. agree only in part with it. According to them the size of the vane will affect the required torque for a given stress but it does not change the development of stress. Thus, the vane dimensions should be immaterial in determining the yield stress.

Yan et al. ([80]) used a commercial finite–element package (FIDAP) to model the behavior of Herschel–Bulkley, Casson and a Maxwell type fluids within a domain of 1300 elements. They assumed a shearing surface located on a cylinder whose dimensions correspond to those of the vane, an uniform shear–stress distributed on this surface being equal to the yield stress and no secondary flows between the vane blades.

Results from Keentok ([48]) are criticized since the way the yield surface is determined is not shown, furthermore, the number of elements between the blades is too coarse (there were only four elements). For this reason, no attempt to investigate the nature of the yield area through elastic, viscoelastic and plastic behavior is made.

Characteristics of the flow such as velocities, streamlines, pressure and shear rate are presented. No difference was found between the Herschel–Bulkley and the Casson model regarding the fluid trapped between the vanes while for the Maxwell model differences were found in the strain rate near the vane tips (which does not agree with the cylinder assumption). For the viscoelastic material there is a more or less uniform distribution of the shear rate away from the tips of the blades. This distribution is explained in terms of the elastic and the viscous part of the shear. That means that the elastic shear is mainly located in the area around the blade tips while the viscous shear is more uniformly distributed along the yield area. The asymmetry in the shear rate distribution found in the numerical simulations is attributed to the effect of elasticity. As a consequence the cylinder assumption would be valid only for certain models, geometry and flow conditions.

Daubert et al. ([28]) related the vane technique when measuring the yield stress to spreadability, a subjective term used in food technology. For a good product, or say, for a spreadable product the yield stress is inverse proportional to the yield stress of the product. Measurements with the vane allowed them to produce "texture maps" easily and avoided disruption of the sample upon immersion of the vane. A total of 13 different products were tested with different vane geometries. To be in agreement with the cylinder assumption they selected radii of the height–to–diameter of the vane greater than 2.0.

Their results of the yield stress measurements indicate that any influence of the end–effects were negligible. They did not use any constitutive models. Paradoxically, some materials that had a low yield stress were able to withstand a large degree of deformation after yielding, suggesting that effects that were not considered in their analysis (e.g. viscosity) play an important role for the vane flow.

Perez et al. ([63]) performed an analysis of the vane geometry with an Arbitrary–Lagrangian–Eulerian (ALE) formulation within a finite element domain consisting of 1492 elements. They presented an analysis of the stress distributions on the shearing surface. Their analysis considered time and size effects, that is, the influence of the rate of rotation of the vane on the surface of shearing. These results show a shearing surface of 1.01 times larger than the vane radius (slightly smaller than that obtained by Keentok et al. ([48])).

Numerical simulations for the 2–D vane geometry depended upon two dimensionless parameters that were represented and limited by inertial and viscous regimes (the last one being typical in vane tests). Before formulating the problem they showed that stress distributions along the top of the vane are not uniform. It was conjectured that material anisotropy and progressive failure are the cause of this non–uniform stress distribution. This suggests that the material model will determine how the stresses are distributed.

When the material is anisotropic the interpretation of the test becomes difficult. For example, the maximum shear can be reached in the vertical surface while the behavior at the top maybe elastic. Since the ratio of both stresses (that at the top and that at the vertical surface) may vary depending on the vane dimensions the influence of the vane dimensions can not be excluded (contrary to the affirmation of Castell–Perez et al. ([23]) and Liddell et al. ([62])). If the contribution of the failure at the top of the vane is dismissed from the total torque (as it has been done usually in practice) then the stress would be underestimated.

Glenn et al. ([36]) tried to obtain device constants for a four–bladed vane in order to obtain averaged shear stresses and shear rates. It was expected that this should lead to the characteristic flow curves of a power–law fluid. The way the constants were obtained was dependent not only on the vane dimensions (again the cylinder assumption is adopted as in [48], [11], [63]) but also on the power–law index n of the material. For the geometries used, the expected behavior is close to the bob–in–cup geometry as n decreases (it is not clear what type of behavior is meant there, probably the characteristic flow curves). Thus, the method used to estimate the device constants would not allow to characterize any type of fluid nor use any vane–geometry due to the dependence upon the type of fluid.

Bravian et al ([15]) used a six–bladed vane with certain slight modifications. For instance, the bottom of each blade was sharpened to reduce sample disruption. To correct deviations from a bob–in–cup system he included a correction factor approximated by a power series. He characterized Newtonian fluids and measured the yield stresses of Power–law fluids in the same manner as carried out by Liddell et al. ([62]).

He noticed the difficulty of defining a shear rate factor due to the non–linearities observed in the curves of shear stress vs. time. Only at the beginning of the linear stress region would it be allowed to introduce a shear rate formulation. The effective radius should be equal to the height of the triangle formed by the tips of two adjacent blades and the corner resulting from the union of the blades.

Rolon–Garrido et al ([69]) studied the non–linear elastic behavior of worm–like micelles in aqueous solutions (cetylpyridinium 100mM / sodium salicylate 60 mM chloride (CPyCl/NaSal) dissolved in distilled water) with a six–bladed vane. This non–linear behavior, characteristic in micellar[3] systems, is represented by a non–monotonic flow curve divided into three main regions. The vane technique helped them to establish conditions when the flow became unstable, a condition that depends upon shear rate. They were able to reach shear rates higher than with a cone–

[3]Micellar solutions can be regarded as dispersions of small particles (usually spheres or ellipsoids).

plate or plate–plate system since for these systems the sample is ejected and slip effects occur.

Farias et al. ([33]) characterized viscoplastic materials with different geometries. Different bob–in–cup systems were used (different heights, lengths, modified surfaces: roughed surface, smooth surface) and compared with a vane geometry (No information is given about how many blades were attached to the shaft). The vane performed better than the cylinder with smooth surface at low shear rates and agreed well with the one with roughed surfaces indicating that it is suitable to eliminate slip–effects. At higher shear rates there were discrepancies between the bob–in–cup systems (roughed and smooth surface) and the vane, discrepancies that were attributed to secondary flows.

Krulis et al. ([49]) approached a four–bladed vane geometry similarly as by Glenn et al. ([36]). They proved that the vane is suitable for food products, analogous to the results of Briggs et al. ([18]).

Another application of the vane for food products was carried out by Martinez–Padilla et al. ([53]). They used the system to characterize two different type of sauces, one that contained fine particles and another with coarse particles. They used two different aspect ratios for the vane–in–cup geometry, one with $\kappa = 1.06$ and a large–gap configuration with $\kappa = 2.0$ [4]. The vane–in–cup with small–gap used four blades while the one with the larger gap had eight blades (in both cases the blades were equally spaced).

Their measurements were in agreement with measurements performed with the bob–in–cup geometry. As previous works, they based their investigations on the solid body rotation thereby employing a Couette analogy for small and large gaps. Curiously they conclude recommending the vane–in–cup with large κ. It is known that there is no viscometric flow in this system and that Couette approximations in the bob–in–cup system get worse as κ grows. They calculated shear rate and shear stress factors based on the material properties of the investigated sauces departing from a Couette flow and used a correction factor for the shear viscosity as proposed by Bravian et al. ([15]).

Fisher et al. ([35]) used a 4–bladed vane to investigate measurement errors that arise in yield–stress rheometry in modern rheometers as a result of an extra induced torque upon immersion of the measuring device into the cup containing the sample. In most cases a residual torque is not negligible and can be still present if consecutive

[4] κ is given by the ratio of the outer cylinder to the inner cylinder radius.

measurements are performed with the same sample. This would lead eventually to erroneous measurements and the tests will not be reproducible. Modern equipment have built–in software to set this initial torque to zero at the beginning of the test to avoid or at least to reduce the effect of this additional torque.

For the calculation of the yield stress they use the methodology proposed by Nguyen et al. ([58]) with a vane having a ratio length–to–radius equal 2.0 (l_v/r_v). If the vane is immersed carefully and no stress is induced, using this modern software–feature to zero the initial stress will make no difference. However, if an initial stress is present and this stress is zeroed, errors will not be eliminated from the measurement and any reproducibility will be difficult to achieve.

2.1 Effect of vane dimensions

Previous approaches seem to fit the experimental data for Newtonian fluids well. Although this may be true, one has to notice that the dimensions of the vane are an important factor that can not be ignored nor should be taken arbitrarily especially when the shear rate is calculated under the assumption of a solid body rotation.

Castell–Perez et al. ([23]) investigated 10 different geometric combinations. In their experiments they kept the diameter of the vane constant while the height of the vane and the ratio vane–diameter to cup–diameter was varied in a certain range. They concluded their work affirming that a certain averaged–constant used to estimate the shear–rate changes as the geometry of the system is modified and / or the material properties change. They used Newtonian and Ostwald de Waele fluids.

An important aspect in the measurement of the yield stress is the minimal influence of the vane dimensions. Liddel et al. ([62]) have performed measurements of the yield stress for different suspensions. In their work they compare the results of other researchers with their results (see for example [58], [83]) to show that the vane dimensions have little effect on the measured yield stress. The size of the vane affect the required torque for a given stress but do not change the development of the stress. This is in agreement with the definition of the yield stress as a material parameter.

On the other hand, for the viscosity, Castell–Perez et al. ([23]) found differences when estimating the constants to compute the shear–rate, that is, the ratio $\kappa = r_o/r_v$ has an influence on the shear rate. Evidently one expects higher shear–rates for lower values of κ. For accurate measurements the ratio length–to–radius of the vane should be greater than 2 ($l_v/r_v > 2$).

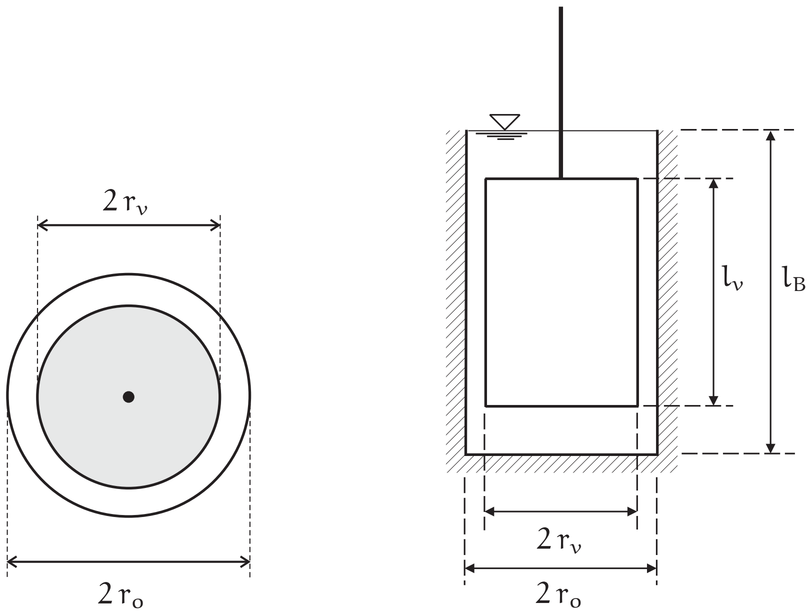
2.2 End effects

In the previous section, works carried out by different researchers have shown how the length of the vane and the outer cup can affect the measurement of the viscosity and that the interpretation of the data may become difficult especially for complex fluids. For a small gap (small κ) these problems can be associated with the ends of the vane (bottom and top). How strong or weak they are will depend on the nature of the fluid, whether it is Newtonian or non–Newtonian, shear–thickening or shear–thinning. It is well known that slip might be a problem when using a Searle or Couette rheometer in a concentrated suspension. In this type of rheometer, the fluid in contact with the bottom contributes a drag which must be matched by the applied torque. The profile of this torque is well described in the annular region (along the whole length l_v of the cylinder) but over the immersed end in an undefined way. If the gap width is kept small enough so that $r_o - r_v << r_o$ then the above problem is reduced since the contribution of the bottom becomes negligible ([66]).

Ideally in these conditions the shear stress at the outer wall is:

$$\tau_o = \left(\frac{r_v}{r_o}\right)^2 \tau_i \, . \tag{2.1}$$

Here τ_i is the shear stress at the inner wall.



(a) Top view. (b) Lateral view.

Figure 2.1: Bob–in–cup geometry.