# 1

# Introduction

This study focuses on espresso coffee extraction. The investigation is divided into the steps involved in the brewing process, from the tamping of the packed bed to the analytical determination of the extraction kinetics from individual key components. The main objective is to develop an experimental and theoretical approach to impact the final cup constitution from controllable parameters directly. The work presented here provides a verified method for improving extraction consistency.

Coffee is one of the most traded and consumed commodities worldwide. Among all the existing brewing methods, espresso is characterized by percolating hot water ($90 \pm 5^{\circ}$ C) through a dense-packed bed of roasted and finely ground coffee under high pressure (Illy and Viani, 2005). Even though regarded as a simple and everyday process, espresso brewing is highly complex; many intercorrelated parameters determine the extraction of the soluble material from the surface and the pores of coffee particles (Petracco, 2001). Apart from the solubilization of the hydrophilic substances, further phenomena occur like the emulsification of coffee oils (Illy and Viani, 2005), suspension of solid coffee cell-wall fragments (Illy and Navarini, 2011), and particle swelling (Mateus and Rouvet, 2007).

## 1.1 Motivation and objectives

The current project was based around fully automated bean-to-cup coffee machines. These are constituted to perform every task that a regular barista would do to obtain a characteristic espresso with the attributes that make it preferable for consumers. These steps, shown in Figure 1.1, include milling the beans, compressing the bed, setting the proper water temperature, percolating the water through the bed, and stopping the extraction at the right moment. Moreover, not only is the final cup profile an essential asset of espresso brewing, but also the distinctive highly viscous slow velocity flow.

The fully automated machine mills the coffee beans on demand, ensuring better conservation of aroma components. Additionally, it offers the opportunity to influence the whole extraction by simply modifying an initial parameter, in this case, the particle size distribution. The milling grade could be automated using mathematical correlations, leading to a personalized final cup on demand.

Nonetheless, such machines also exhibit disadvantages that compromise the final cup quality. For instance, some fully automated machines store the beans in a hub to mill on-demand when the next customer selects an espresso-based drink. Dosing,

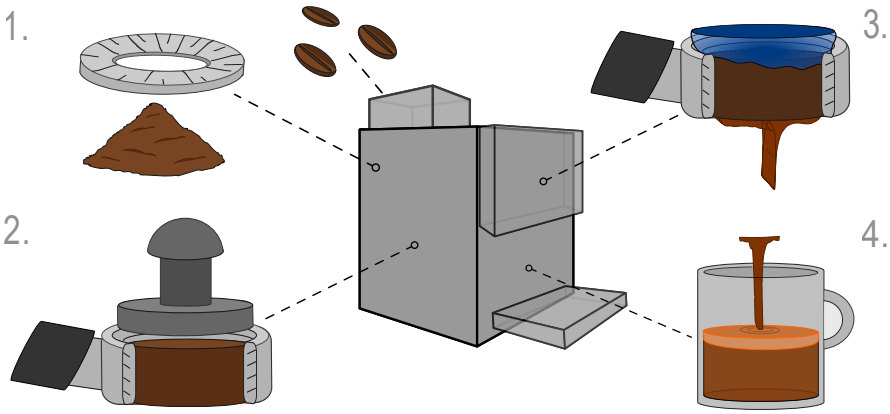


Figure 1.1: Schematic representation of tasks performed by a fully automated machine to brew coffee: 1. milling coffee beans, 2. packed bed compression, 3. water percolation through the bed, 4. extraction kinetics and final cup size.

compression of the bed (tamping), and stopping the water pump, defined by the settings automatically, also show slight variations between cycles when brewing. These fluctuations are caused mainly due to three reasons:

1. The beans on the machine hub are open to the surroundings without an effective barrier against moisture uptake or oxidation even when the hub is enclosed (as shown in Figure 1.1). The longer the beans remain in the hub, the more probable they experience structure modifications that will impact the extraction process.

2. The machine milling blades can get misaligned or heated up from friction, which brings variations to the resulting particle size distribution, and therefore, to the whole extraction.

3. Discrepancies in the extraction process's crucial steps, like dosing, bed compression, or pump regulation, can cause variations in the extraction conditions, namely volume flow and pressure drop, and, therefore, in the final cup profile.

This work aims to evaluate the impact of the parameters that fully-automated machines could directly control, namely from Figure 1.1 the 1) particle size distribution and 2) bed compression on 3) extraction process conditions, and 4) final cup profile, establishing a correlation between the main parameter of particle size and the successive steps in espresso brewing. The approach would enable the optimization of other parameters, reducing the impact of small fluctuations and providing the best quality of a specific final cup. The best approach to integrating these correlations is employing a mathematical and mechanistic model. Such a model would be advantageous for designing future machines adapted to control those crucial parameters, as identified by the modeling results, to counteract variations in the cup quality.

The first task in the extraction process consists of milling the roasted coffee beans. Many parameters impact this process; for instance, a higher roasting degree leads to a more brittle structure of the matrix, usually causing a finer particle size distribution. The particle size is relatively easy to modify compared to other parameters, even for fully automated machines. However, obtaining a precise and consistent

particle size distribution is not trivial. Therefore, an essential condition adopted in each extraction experiment was the meticulous control of the particle size distribution. This way, the real impact of this parameter can be quantified and correlated with the results. The correlations with other parameters are essential to predict the outcome and effect of particle size variations and thus modify these to regulate and counteract the fluctuations in the particle size.

## 1.2 Fundamentals of espresso extraction: state of the art

Studying and modeling coffee as a food system is challenging, mainly because of the complexity of material parameters like compositions, geometry, phase transitions, etc. (Fries, 2021). Coffee particles are a complex cellulosic matrix with internal porosity and large variability of surface geometries far from perfectly smooth spheres (Petracco, 2005b).

Espresso brewing is distinguished from other coffee methods because it uses a high-pressure driven flow through a tightly compressed packed bed of finely milled coffee particles. A slow and pulsing pace characterizes the volume flow. Hence, the obtained cup is relatively small, ranging from 30 to 40 ml, with a high concentration of extracted solids (Parenti et al., 2014). The short cup beverage obtained is also a highly complex system constituted by an enormous number of volatile and non-volatile components. One can consider the brew an emulsion from the different oils and a suspension from the finest particles collected by the hot water percolating through the bed (Petracco, 2005a).

As shown in Figure 1.1, the steps involved in the brewing of espresso take place in a specific sequence: milling the coffee beans, compressing the packed bed (also known as "tamping"), percolating water through the milled coffee while rising the pressure drop across the packed bed for the water flow, and producing the correct final size of the cup by stopping the water pump. The different process parameters involved in these steps intercorrelate considerably, and studying their independent correlations results in a significant challenge (Petracco and Suggi Liverani, 1993). Therefore, to develop a mathematical correlation encompassing the macroscopic phenomena involved before and during bed percolation and extraction, requires a systematically evaluation of the main parameters. For this purpose, experiments were carried out in each espresso step.

### 1.2.1 Characterization of bimodal distributions

Ground coffee particles have a bimodal distribution (Petracco, 2005a). The fines fraction, the volume proportion of particles measuring less than 100 $\mu$m, can significantly reduce bed permeability during water percolation. The latter is mainly because of their large flowability, causing the clogging of the bed pores. This particle volume fraction also possesses a notably high specific surface area, directly influencing the extraction process.

To characterize the bimodal particle size distribution (PSD), the Rosin-Rammler model was fitted to the coarse fraction ($d_p > 100\ \mu$m) in each cumulative distribution fraction curve. The model is a two-parameter exponential function given by Vesilind (1980) as follows:

$$Q_3(x) = 1 - e^{\left(-\frac{x}{\eta}\right)^{\gamma}} \tag{1.1}$$

where $Q_3$ (%) is the cumulative volume fraction, $x$ is the particle size, $\eta$ is the volumetric mean particle size, and $\gamma$ is the size uniformity factor, which characterizes the range of particle sizes in the distribution. A lower value of $\gamma$ indicates a less monodispersed distribution; hence, a larger polydispersity of the collective particle sizes is expected.

### 1.2.2 Particle porosity of roasted coffee

The method adopted to determine the packed bed porosity is reported in Corrochano et al. (2015). The internal porosity in coffee beans depends on their roasting degree. Hence, the density of the particles $\rho_{part}$ is calculated given the intrinsic solid density of the coffee matrix $\rho_{solid}$ and the particles internal porosity $\varepsilon_{particle}$, as shown in Equation 1.2:

$$\rho_{part} = \rho_{solid}\ (1 - \varepsilon_{part}) \tag{1.2}$$

where $\rho_{solid}$ refers to the mass of coffee divided by its solid matrix volume, thus the intrinsic solid density of the roast coffee, and $\varepsilon_{part}$ is the porosity of the particles. The measured total (skeletal) porosity of a sufficiently small particle size distribution is unlikely to contain any closed pores (Corrochano et al., 2015).

The intact cell pockets in a given coffee particle have a diameter of $25 - 40$ $\mu$m (Schenker et al., 2000). Based on this definition, the authors assume that the largest possible pore size within a particle is 40 $\mu m$ (Corrochano et al., 2015; Melrose et al., 2018). When using a mercury porosimetry method, this pore size is measured by the amount of applied pressure with which mercury penetrates pores within particles.

## 1.3 Extraction kinetics

Espresso composition contains more than 800 components (Petracco, 2001). Therefore, selecting components with specific properties is crucial to achieving a general conclusion from the analysis. Moreover, the study of extraction kinetics is relevant for determining and controlling the final cup sensory profile. Recent studies correlate this sensory profile with the concentration of specific components and underline the relevance of measuring them separately from the total extracted solids. These key marker components, like caffeine and trigonelline, contribute to the final bitterness (Belchior et al., 2019; Maeztu et al., 2001), whereas compounds like carboxylic and chlorogenic acids are associated with acidity (Esteban-Díez et al., 2004). Parenti et al. (2014) state that their solubility drives the extraction efficiency of the coffee constituents in water. Decreasing the particle size leads to higher simultaneous extraction of caffeine and trigonelline. The diffusion process of these components in the swollen particles is the limiting step during extraction. These findings are precise since particle size distribution of the coffee powder has a direct impact on both the convection and the diffusion process occurring during extraction (Kuhn et al., 2017; Spiro and Selwood, 1984; Zanoni et al., 1992). Moreover, de Vivo et al. (2022) studied the impact of different particle size distributions on the extraction of volatile compounds in espresso. The authors found that the highest extraction of most of the studied volatile compounds was obtained using particle size distribution with size fractions between 300 and 425 $\mu$m.

Some studies develop and validate extraction models for aroma extraction kinetics by classifying the compounds based on their extraction kinetics. The authors state that for extracting components with low solubility (strongly non-polar components), the particle surface area is the limiting factor (Beverly et al., 2020). They propose larger concentration gradients between intra and intergranular pores to enhance mass transfer. The temperature of the extracting water also greatly impacts the extraction kinetics of the espresso components, as characterized and modeled by other authors (Navarini et al., 2009; Pannusch et al., 2023).

### 1.3.1 Lipids

Lipids are an important extractable component in espresso brewing. According to some authors, the presence of lipids affects the perception of the final cup acidity, since they act like a blocking interphase for the receptors in the human tongue (Petracco, 2001). The emulsification process of the lipids are also responsible for the characteristic viscosity of the brew. In this work an analytical study was done to determine the amount of lipids which are contained in the surface of the particles as function of the milling degree.

## 1.4 Mechanistic model to describe the espresso extraction process

The proper way to understand all the processes occurring during espresso extraction is through mathematical correlations. The central core of the processes occurs as soon as the percolating water contacts the packed bed. A mass transfer process of the soluble components occurs from the particles to the flowing water. As previously described, coffee particles consists of a complex matrix with internal pores; therefore, the dominating mechanism is a diffusive mass transfer. Simultaneously to this process, the advected flowing water causes a convection mass transport of the components through the bed. The coffee particle sizes, which determine the diffusive length and the volume flow velocity, directly impact both processes. Therefore, the particle size determines the extraction kinetics from components.

Published works have been dedicated to describing the extraction process, especially regarding optimizing the brew quality (Ellero and Navarini, 2019; Moroney et al., 2015), which has been proven to be related to the extracted compound composition in the final cup (Navarini et al., 2009). Several studies have confirmed that the water temperature and particle size distribution, among all the other variables, predominantly impact the extracted component compositions. Lower extraction yields associated with under-extraction are related to an acid or sweet sensory profile, whereas higher extraction yields or over-extraction result in an astringent bitter profile (Petracco, 2001).

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According to relevant coffee extraction studies, the main parameters and process conditions that broadly impact the kinetic extraction of studied components are:

1. Particle size: the microstructural restrictions and caffeine forming complexes with other compounds (Spiro and Selwood, 1984).
2. Hydrodynamics of the system: effects of the external boundary layer are negligible; instead, diffusion is the primary limiting step inside the grains (Spiro and Page, 1984).
3. Hindrance factor: the ratio of the bulk to the estimated diffusion coefficient. That is the microstructural correction factor (Del Valle and de La Fuente, 2006). Roast and ground coffee is a hydrophilic material and water penetrative. Therefore, the finite time required for the water to ingress and dissolve the components, hence the water inflow hindering the outflow of caffeine, is expected to be relatively short. The decrease in the hindrance factor is primarily due to modifications of the microstructure due to swelling.
4. Temperature: the increase in the hindrance factor is attributed to a lower dissolution rate of caffeine at lower temperatures (Spiro and Chong, 1997).

Apart from espresso extraction, many other analogous studies focus on packed bed reactor systems containing organic swelling granular material and model approaches for process optimization (Alaqqad et al., 2012; Arora and Potůček, 2009; Brodin et al., 2013; Potůček and Miklík, 2010; Reynolds et al., 2015). A dispersion model is employed in all these studies to accurately describe the non-ideal flow in biomass-packed beds.

## 1.5 Microscopic scale: mass transfer from single particles

The mass transport mechanism governing the extraction of soluble components in the coffee particles is described by the Fick's first law (Crank, 1975):

$$J = D_b \left( \frac{\partial C}{\partial x} \right) \tag{1.3}$$

where $J$ ($\mathrm{kg/m^2\,s}$) is the diffusion flux, $D_b$ ($\mathrm{m^2/s}$) is the bulk diffusion coefficient, and $(\partial C/\partial x)$ ($\mathrm{kg/m^4}$) is the concentration gradient. In solid-liquid extraction processes multiple effects may hinder the diffusion due to properties of the solid phase. The solid phase effects with an impact on the diffusion are:

- The finite time required for the liquid to ingress in the solid and dissolve the solute from the solute matrix.
- A possible reabsorption of the solute in the solid matrix or interactions with other co-solutes (Schwartzberg and Chao, 1982).
- Diffusion length modification due to swelling or erosion of the polymeric matrix (Mateus and Rouvet, 2007).
- The microstructural effects caused by a combination of hindering effects of the surface and cavities inside the particles pores.

- The longer paths for diffusing components molecules caused by tortuosity.

Therefore, an effective diffusion coefficient is employed instead of a bulk diffusion coefficient. Due to the non-steady nature of the solid-liquid extraction, the time-dependent concentration gradient of a given species inside the solid comes from Fick's second law. For spherical coordinates the corresponding equation has the following form Crank (1975):

$$\frac{\partial C_s}{\partial t} = D_{eff}\left(\frac{\partial^2 C_s}{\partial r^2} + \frac{2}{r}\frac{\partial C_s}{\partial r}\right) \tag{1.4}$$

where $D_{eff}\,(\mathrm{m}^2/\mathrm{s})$ is the effective diffusion coefficient, $C_s\,(\mathrm{kg}/\mathrm{m}^3)$ is the concentration of the solute inside the solid, and $r$ (m) is the radial coordinate. Some approaches to determine the effective diffusion coefficients involve extraction simulations and experimental data. Various studies are dedicated to kinetics studies of overall extraction or specific components, mainly caffeine, as previously described in section 1.3.

Moreover, the total macroscopic flux from the inside of the particle kernels through the internal pore space to the bulk of flowing water around the particles is an average of molecular diffusion and the dispersive flux for espresso extraction (Moroney et al., 2015). As the internal pores structure directly influences the length of the diffusive path, the diffusion coefficient is a function of the internal porosity of the particle. In the expression adopted from (Millington, 1959):

$$\Theta = (\varepsilon_{part})^{-1/3} \tag{1.5}$$

tortuosity $\Theta$ (-) is the ratio between the actual path length and the macroscopic path length. Given that in isotropic porous mediums, the effective molecular diffusion coefficient is defined as $D/\Theta$, it can be then incorporated into the total intergranular mass transport:

$$\mathbf{A} = \alpha\, S_1\, (\varepsilon_{part})^{4/3}\, (C_v - C_h) \tag{1.6}$$

where $\alpha$ (m/s) is the mass transfer coefficient, $C_v$ and $C_h\,(\mathrm{kg}/\mathrm{m}^3)$ refer to the intragranular and intergranular averaged concentration respectively, and $\varepsilon_{part}$ is the particles porosity. The difference between $C_v$ and $C_h$ is accounting for the driving force of the diffusive mass transfer. $S_1(\mathrm{m}^2/\mathrm{m}^3)$ represents the specific surface area.

The diffusion from the particles' surface is a solubilization process that involves several complex phenomena. Existing available models assume that all the soluble mass on the surface (represented as the volume fraction $\phi_{s0}$) is already solubilized at an infinitesimal distance next to the solid surface. This solution already reaches the equilibrium; therefore, the concentration is considered the saturation concentration $C_{sat}$. The occurring mass transfer is an interphase mass transfer process, where the component molecules diffuse to the bulk flow (Moroney et al., 2015), as described in Equation 1.7:

$$\mathbf{B} = \beta\, S_2\, (\, C_{sat} - C_h) \tag{1.7}$$

where $\beta$ (m/s) is the mass transfer coefficient, $C_{sat}$ is the saturated concentration of soluble coffee in the matrix of the particle and $C_h$ is the intergranular concentration of the bed, and $S_2$ $(\mathrm{m}^2/\mathrm{m}^3)$ is the specific surface area, where the extraction takes place.

## 1.6 Macroscopic scale: packed bed of coffee particles

The tamping process compresses the coffee particles inside the portafilter with a piston-like tool with the same diameter as the bed. Usually, a tamping force from $30x10^3$ up to $70x10^3$ Pa (130 to 200 N) is employed to compress the bed of 60 mm portafilter (Petracco, 2005a). This tamping force must be sufficient to provide the right initial bed porosity conditions to achieve the characteristic low volume flow rate during extraction. The wide range of particle sizes (20 $\mu$m to 490 $\mu$m range for espresso extraction) and the deviation from ideal spheres allow the particles to form a densely packed arrangement in the portafilter bed when compressed.

The influence of particle size polydispersity on the granular packing under compression is an object of several studies for different granular materials. Annabattula et al. (2012) observes the micromechanical behavior of binary and polydispersed spherical pebbles using a DEM approach and finds that mono-sized assemblies show a larger resistance to compression compared to binary and polydispersed assemblies. Sohn and Moreland (1968) report that packing densities of binary mixtures increases with the mean size ratios of the components. The authors claim a maximum packing density value at 55 to 75% of the larger component fractions. Wiacek and Molenda (2014) demonstrate that important mechanical properties of granular material, like the coordination number, decreased with polydispersity. In contrast, the expansion of granular bulk in the perpendicular direction to the compression tended to increase.

### 1.6.1 Compression of granular beds

To determine the bed bulk density of the packed bed, the correlation of it with the porosity of the particles is defined as follows:

$$\rho_{bulk} = \rho_{part}\ (1 - \varepsilon_{bed}) \tag{1.8}$$

where $\rho_{bulk}\,(\mathrm{kg/m^3})$ is the bed bulk density, $\rho_{particle}\,(\mathrm{kg/m^3})$ is the density of the roasted coffee, and $\varepsilon_{bed}\,(-)$ is the porosity of the packed bed.

An empirical mathematical expression is used (see Equation 1.9) to model compressible packed bed reactors with different granular materials (Verhoff and Furjanic Jr, 1983):

$$\varepsilon_{bed} = \varepsilon_0\, e^{-\omega\sigma} \tag{1.9}$$

alternatively, it can be expressed as equation 1.10:

$$\varepsilon_{bed} = \frac{\varepsilon_0}{1 + \omega\sigma} \tag{1.10}$$

where $\varepsilon_{bed}\,(-)$ is the obtained bed porosity from compression, $\varepsilon_0\,(-)$ is the initial bed porosity resulting from the powder in repose, and $\omega$ (1/Pa) is the intrinsic compression factor. These are constants obtained from experimental data for different granular materials. The bed axial compression stress is the variable $\sigma$ (Pa). These parameters obtained experimentally assume that the decrease in bed volume as a function of pressure is due to loss in void volume (Verhoff and Furjanic Jr, 1983).

The container wall considerably influences the compression of the packed bed, acting as a resistance force as a function of compression axial stress. This parameter is

intrinsic to every granular material and will depend on the diameter of the container (McCabe and Smith, 1976).However, the wall effect is neglectable if the bed-particle diameter ratio is larger than 5, assuming a homogeneous bed (Di Felice and Gibilaro, 2004). Other factors also impact granular compression, mainly related to the particle size distribution width and micromechanical properties (Azéma et al., 2017).

The theoretical principles of the bulk porosity from spherical particles bimodal distribution of spherical characterized by Brouwers (2006) describe the compression behavior of such size distribution. The authors developed a model to predict the minimum attainable bulk porosity depending on the large and small particle fraction and the corresponding size ratio of the bimodal distribution of granular materials.

## 1.7 Mechanics involved in the espresso packed bed

A force balance across the length of the packed bed (considering $z$ the axial coordinate and $z = 0$ the top of the packed bed) allows for the calculation of the axial tension as function of the bed length. The schematic representation of the acting forces in the packed bed portafilter is shown in Figure 1.2. The sum of the total forces across the bed length is then defined as:

$$\Delta F = F_f - F_w + F_g \tag{1.11}$$

where the hydrodynamic force, represented as $F_f$, results from the fluid flow through the bed, where the force is related to the pressure drop through the cross sectional area of the portafilter and is described by Darcy´s law (Corrochano et al., 2015):

$$F_f = A\,\Delta P = \frac{\mu\, Q}{K}\Delta z \tag{1.12}$$

and across the bed height can be then rewritten:

$$Q = \frac{K A}{\mu\, L}\Delta P \tag{1.13}$$

where Q (ml/s) is the volumetric flow rate, A ($m^2$) is the cross-section area in the axial direction, $\mu$ (Pa s) is the fluid viscosity , $\Delta P$ (Pa) is the pressure drop across the bed, and $K$ ($m^2$) is the permeability of the porous medium. Darcy´s law is valid for low Reynolds numbers ($Re_p < 10$), where the relevant length scale concerns the pore size of the bed.

In the case that the flow goes from the top to the bottom of the bed, the gravitational force $F_g$, which is a function of the packed bed weight, acts in the same direction, defined as:

$$F_g = A(\rho_s - \rho_w)\, g\, \Delta z \tag{1.14}$$

where $\rho_w$ ($kg/m^3$) is the density of the flowing water, $g$ ($m/s^2$) is the gravity acceleration. The last considered force $F_w$ is the wall friction force that acts as a resistance to the two other forces. This force is dependent of the portafilter cross sectional area, $\mu_w$ the friction coefficient and $\nu$ the horizontal load ratio, portafilter diameter and material-dependent empirical parameter, or Poisson's ratio as follows:

$$F_w = \frac{\nu\, \mu_w D_p\, \pi}{A} F\, \Delta z \tag{1.15}$$

where $D_p$ is the diameter of the portafilter, and F (N) is the resulting force from the wall friction along the height of the bed. From Equations 1.13, 1.14, 1.15 , the force balance in Equation 1.12 can be written as the resulting axial pressure across the bed:

$$\frac{\partial P}{\partial z} = \frac{1}{A}\frac{\partial F}{dz} = \frac{\mu Q}{KA} - \nu\mu_m P + (\rho_s - \rho_w)g \tag{1.16}$$

In an espresso packed bed in a portafilter, the gravitational force $F_g$ can be neglected due to the relatively low mass used for every espresso cup. Moreover, the wall friction force in the espresso portafilter can be neglected since the bed particle diameter $\geq 5$ (Corrochano et al., 2015).

Accordingly, the Euler dimensionless number ($Eu$ number) is used in hydrodynamics to express the relationship of pressure drop caused by a flow resistance and the inertial forces of the fluid, as defined in Equation 1.17:

$$Eu = \frac{\Delta P}{\rho_w \mathrm{v}^2} \tag{1.17}$$

where $\Delta P$ (Pa) is the pressure drop over the bed, $\rho_w$ (kg/m$^3$) is the density of the flowing water and v (m/s) the interstitial velocity, defined in Equation 1.18 as:

$$\mathrm{v} = \frac{u}{\varepsilon_{bed}} \tag{1.18}$$

where v (m/s) is the interstitial velocity and $u$ (m/s) is the superficial velocity.

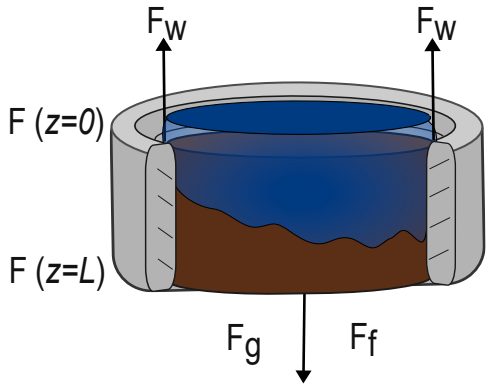


Figure 1.2: Balances of the forces acting in the packed bed portafilter during water percolation. $F_w$ refers to the wall friction force from the portafilter, $F_g$ is the gravity force acting on the bed and $F_f$ is the inertial force from the fluid volume flow through the bed. $z = 0$ is the top of the bed and $z = L$ the bottom.

### 1.7.1 Coffee bed permeability

During the percolation process, the highly dense bed directly affects the bed permeability reported in a range of 3x10$^{-14}$ to 8x10$^{-13}$ m$^2$ (Corrochano et al., 2015). Hence, water percolation through the packed coffee bed is a complex dynamic process with microscopic phenomena occurring simultaneously until a final bed porosity