Research Article

ISSN 2689-1050

Chemical & Pharmaceutical Research

Hydrodynamics in Packed Beds with Particular Focus on UHPLC Columns

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Received: 11 Apr 2025; **Accepted:** 20 May 2025; **Published:** 28 May 2025

Citation: Quinn HM. Hydrodynamics in Packed Beds with Particular Focus on UHPLC Columns. Chem Pharm Res. 2025; 7(1): 1-10.

ABSTRACT

*Packed beds have been in use for a variety of applications for more than 100 years. Today, they have grown in importance with the emergence of new technologies like the need to generate better ways to store thermal energy in support of renewable sources such as wind, sun and other green energy types. The use of rigid particles has been, arguably, the most versatile packing materials across a broad spectrum of applications. However, to be thorough, one must consider all three categories of particle porosity which make up this broad field. These categories are, (1) nonporous particles ($\varepsilon_p = 0$), (2) partially porous particles ($0 < \varepsilon_p < 1$) and (3) fully porous particles ($\varepsilon_p = 1$). Foremost amongst all applications, perhaps, has been the field of HPLC (**H**igh **P**ressure **L**iquid **C**hromatography) which, for the most part, utilizes category 2, i.e., partially porous particles, which morphed out of the original field of Gas chromatography. The use of smaller and smaller particle diameters to achieve ever increasing separation efficiencies, however, has generated the need for a comprehensive assessment of the hydrodynamics of packed beds. As particle diameter decreases, the operating pressure drops increase, necessitating higher packing pressures which can result in particle compression when the particles are not sufficiently rigid. In the case of partially porous particles, increased packing pressures can also lead to a reduction in internal particle pore volume. This new assessment requirement dictates the need to develop a unified framework which can seamlessly describe the fluid dynamics of packed beds containing all three categories of particle porosity. In this paper, the goal is to present such a unified methodology with particular emphasis on establishing the fit between the general model and published works, which includes all categories of particle porosity especially those involving the relatively recent development in HPLC columns known as UHPLC (**U**ltra **H**igh-**P**ressure **L**iquid **C**hromatography).*

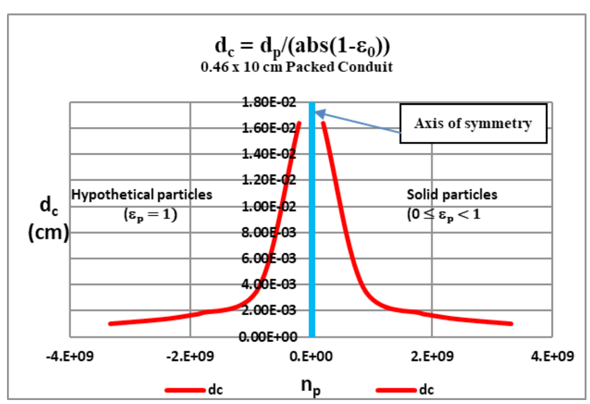
Keywords

Permeability, Particle Porosity, Hypothetical Particles, Forchheimer coefficients.

Highlights

- Flow embodiments in closed conduits contain particles with solid skeletons or free space.
- Dimensionless Permeability identifies the impact of wall effect.
- A unified model defines the universal permeability constants k_1 and k_2.

Graphical Abstract



Graphical Abstract: The diameter of the Hypothetical Q-Channel (d_c) is defined by two distinct types of Particles, (1) Solid particles and (2) Hypothetic particles, each a mirror image of the other. The axis of symmetry is provided by the line representing $n_p = 0$.

Introduction

The hydrodynamics of packed beds of particles has undergone much revision over the last 150 years but much of it has been formulated without the requisite empirical support.

The Kozeny/Carman equation, for instance, is one that applies to permeability in laminar flow and is used extensively in HPLC publications [1]. In 1937, P.C. Carman's publication declared that the "constant" in the Kozeny/Blake equation should be set at the value of 180 [2,3]. It offered as support for this teaching two pieces of evidence which is not transparently obvious. Firstly, the Coulson thesis was cited as supporting empirical evidence [4]. However, on close inspection of this thesis, a meritorious argument can be made that Carman's value of about 180 for the constant in Kozeny/Blake corresponding to Coulson's data was not *objectively* derived. Secondly, in the addendum to the original publication in 1937, it was suggested that as viewed by the naked eye, on a molecular level, fluid moved through the packed bed at a 45-degree angle, an observation that was extrapolated to invoke the theorem of Pythagoras in establishing a tortuosity computation. This methodology is subjective and difficult to replicate.

Georges Guiochon is an author of chromatographic literature whose teaching regarding this value also falls short of universal certification [5]. The parameter, $k_{0,}$ shown at page 153 of this textbook, was defined in combination with mobile phase velocity, to represent the permeability constant of proportionality in laminar flow. This teaching was based upon the use of irregularly shaped particles, without the degree of irregularity being specified. Additionally, because the superficial fluid velocity, which is independent of packed bed total porosity, was not used, this teaching cannot be applied, with confidence, for prediction purposes. Because this teaching involves many examples and is, therefore, beyond the scope of this paper, a detailed evaluation of that teaching is provided [6].

Sabri Ergun, in engineering literature, wrote a series of papers in which the value of 150 was identified for this parameter [7,8]. This value has not stood the test of time either and has been rejected by many investigators since its publication in 1952 [9]. Similarly, in the popular chemical engineering textbook, Transport Phenomena, two instances are provided in which the authors, Bird, Stewart and Lightfoot, did not disclose underlying empirical evidence [10]. Although, in the original edition of the textbook (1960), the value of 150 is based upon an "analysis of a great deal of data", no algorithm is disclosed to balance the measured pressure drop in datasets against calculated values for the Kozeny/Blake equation. Furthermore, in the 1960 edition, the authors stated that a value of 25/6 was used as a modification factor to account for packed beds and applying this factor in the equation, a value of 150 was identified. In the 2002 edition of the textbook, however, a value of 100/3 is substituted for the coefficient of 16 in the application of the Fanning friction factor. This methodology results in the same expression for the Kozeny/Blake equation as well as the same value of 150. Without further information as to the derivation of these vulgar fractions, it is, of course, impossible to productively evaluate such a conclusion. Since the authors arrive at the same destination via two different pathways, however, both of which are unsupported, this teaching does not stand on its own merit.

Finally, we underscore the notable exception in the published chromatographic literature of the teaching of J.C. Giddings, an engineer, interestingly, who dedicated his entire professional life to separation science [11,12]. In the 1965 textbook at page 209, for instance, Table 5.3-1 contains a comprehensive teaching for both porous and nonporous particles, unrivaled in the published literature, which stands as the gold standard for permeability in packed conduits, in the laminar flow regime, even to this very day. Because this teaching is voluminous and complex and, therefore, beyond the scope of this paper, a detailed description of that teaching is provided [13].

In this paper, a novel approach to packed bed hydrodynamics is presented wherein particle porosity is the independent variable which defines a particular packed conduit under study. This teaching refutes the notion that the Kozeny/Carman constant is a variable and dictates that its value is 268.19 approx. over the entire fluid flow regime. This includes the laminar flow regime wherein most UHPLC columns reside.

Methodology

The abbreviation, QFFM, stands for the **Q**uinn **F**luid **F**low **M**odel, which is a comprehensive novel theory of fluid flow in closed conduits. It was published in the year 2019 [14]. This model establishes a unified mathematical platform for all three categories of particle porosities by the imposition of boundary conditions within an all-inclusive porosity function framework. In this paper, examples of packed conduits containing all three categories of particle porosities are used to demonstrate the utility of applying the QFFM to explain the hydrodynamics of fluid flow through closed conduits, across the entire fluid flow spectrum from creeping to fully turbulent flow. In particular, examples of UHPLC columns are chosen which contain the so-called sub-2micron diameter particles, and which are at the forefront of modern-day engineering capability relative to particle size control.

In evaluating any dataset within the context of the QFFM, all measurements of flow rate and pressure drop are accepted as valid. This conclusion is based upon the broadly accepted notion that volumetric flowmeters and pressure transducers are highly accurate when properly calibrated. On the other hand, the measurements of particle diameter and packed column external porosity are universally regarded as fraught with problems. This is particularly true when the particles are very small, compressible and not completely spherical. The teaching of the QFFM is then used to back-calculate the values for the average spherical particle diameter equivalent, d_p, as well as the packed column external porosity, ε_0. These two variables are not independent, but their values must be justified, simultaneously, as dictated by the continuity equation, for any combination of measured values of flow rate and differential pressure. In addition, these variables are used in combination to define the diameter of the Hypothetical Q Channel (HQC) which defines the hydrodynamics in all closed conduits including HPLC and UHPLC columns. Exactly how this is accomplished in the context of the QFFM will now be explored.

Definition of Parameters

The QFFM teaches that there are 17 important parameters in the pressure flow relationship in closed conduits, representing 3 distinct categories of parameters which include: (a) constants, (b) independent variables and (c) dependent variables:

a. There are 4 constants: π, r_h, k_1 and k_2

b. There are 9 independent variables:
- 3 Fluid variables: η, ρ_f and q.
- 4 Packed conduit variables: D, L, n_p, and k.
- 2 Particle variables: d_{pm}, Ω_p.

c. There are 4 dependent variables:
- 1 Fluid variable: $\lambda = f(\pi, r_h, R_{em}, k, \delta,)$
- 3 Packed conduit variables:
  - $d_p = f(d_{pm}, \Omega_p)$,
  - $\varepsilon_0 = f(\pi, D, L, d_p)$,
  - $\Delta P = f(\lambda)$

Where $r_h = 4$ is the normalization coefficient for fluid drag, n_p = the number of particles of diameter d_p in any packed conduit under study, k = the sand roughness coefficient after Nikuradze, d_{pm} = the nominal particle diameter and $_p$ = the particle sphericity (a normalization coefficient for particle shape in the context of the QFFM).

Formula

The QFFM formula can be written as:

$$P_Q = k_1 + k_2 C_Q \quad (1)$$

which is a dimensionless manifestation of what is referred to as Quinn's Law of fluid dynamics in closed conduits. It is a unique formula which combines the above identified variables in a manner not contemplated heretofore.

Underlying Theory

What makes the QFFM unique is that it contains many parameters not identified in other fluid dynamic models, i.e., r_h, k_1, k_2, β_0, τ, λ, Q_N, C_Q, etc., etc., and, in addition, combines all the parameters in a unique arrangement not heretofore available in any other fluid model. In addition, it is the only fluid model that recognizes two additional flow parameters beyond the modified Reynolds number, R_{em}, i.e., δ and λ, which need to be normalized, in order to achieve a unified dimensionless platform of comparison, i.e., the parameter C_Q. (It is recommended that the reader consult the original QFFM publication for all nomenclature and proof of concepts which are beyond the scope of this paper). Thus, when the fluid flow rate, q, pressure drop ΔP, and conduit diameter, D, are determined by experiment and, accordingly, the Forchheimer values of a, and b, are known, the Navier-Stokes equation equivalent may be solved using the QFFM [13]. The accuracy of the solution is driven by accurate measurements of these three variables over a broad range of flow rates, including the non-linear region, where kinetic contributions to measured pressure drop are significant. The solution also involves, necessarily, the fluid property of kinematic viscosity. The procedure to accomplish this is as follows:

The Forchheimer equation may be written as:

$$\frac{\Delta H}{L} = a\mu_s + b\mu_s^2 \quad (2)$$

Where, a, and b, are the Forchheimer coefficients for the viscous and kinetic contributions, respectively, and μ_s is the superficial fluid velocity also referred to as fluid flux. Thus, it is apparent from equation (2) that [$i = \Delta H/L$], known as the hydraulic gradient, is a quadratic function of fluid flux μ_s. It is customary in engineering circles to make a plot of equation (2).

The definition for a, and b, as taught by the QFFM is as follows:

$$a = \frac{4r_h^3\pi\delta\eta}{3\rho_f g d_c^2} \quad (3)$$

and

$$b = \frac{\delta^2\lambda}{2\pi g d_c} \quad (4)$$

It follows from equation (3) above that one may write:

$$\frac{\delta}{d_c^2} = \frac{3a\rho_f g}{4r_h^3\pi\eta} \quad (5)$$

Similarly, it follows from equation (4) above that one may also write:

$$\frac{\delta^2}{d_c} = \frac{2b\pi g}{\lambda} \quad (6)$$

Therefore, in order to solve the N-S equation both equations (5) and (6) must be solved simultaneously.

From equation (5), it is assumed that:

$$\frac{\delta}{d_c^2} = \alpha \quad (7)$$

From equation (6), it is also assumed that:

$$\frac{\delta^2}{d_c} = \beta \quad (8)$$

A further assumption is:

$$x = \alpha\beta \quad (9)$$

and

$$y = \frac{\alpha}{\beta} \quad (10)$$

It then follows that the solution to the N-S equation for closed conduits is:

$$d_c = \frac{1}{x^{(1/6)}\, y^{(1/2)}} \quad (11)$$

$$\delta = \frac{x^{(1/6)}}{y^{(1/2)}} \quad (12)$$

The above simultaneous solution, i.e., equations (11) and (12), for the values of $d_c = d_p/(1-\varepsilon_0)$ and $\delta = 1/\varepsilon_0^3$, respectively, constitutes the solution to the Navier Stokes equation equivalent. This solution depends, not only, upon the independent variables identified above, but also, upon the value of λ in equation (4). However, λ, in turn, although it is an independent variable in the above solution of simultaneous equations, it also depends upon the value of other variables including d_c, a dependent variable itself and, accordingly, and problematically, this is the conundrum of solving the N-S equation. In order to circumvent this conundrum, however, the QFFM defines the value of λ independent of permeability measurements, but, rather, derives it based upon the physics of wall-effects.

In the Supplemental Materials which are part of this publication, the teaching of Giddings is highlighted because it represents teaching for laminar flow applications which predates the QFFM general model and because the latter extends into the realm of very high modified Reynolds numbers, a continuity of methodology is warranted over the entire fluid flow regime.

Data Analysis

The analysis consists of establishing correlation achieved when using this methodology between the measured dataset selected from the literature and the calculated data based upon the QFFM.

There is virtually an exact correlation established for each dataset, because the QFFM uses the Forchheimer coefficients in the manner described below.

Permeability

As can be seen from Figure 1, 9 datasets are presented for packed columns from the published literature which span a broad range of measured flow rates and differential pressure [16-23,]. These examples contain particles with solid skeletons, both nonporous ($\varepsilon_p = 0$, Kang, Buckwald, Erdim) and partially porous, including the UHPLC examples ($0 < \varepsilon_p < 1$, Neue, Gritti, Cabooter). The coordinates shown in the plot are log-log to facilitate a landscape view.

Hydraulic Gradient

The QFFM methodology is based upon the Forchheimer model which balances the measured and calculated data using a quadratic relationship between hydraulic gradient, $i = [\Delta P/(\rho_f gL)]$ and fluid superficial velocity $\mu_s = [4q/(\pi D^2)]$. The linear and quadratic coefficients of the 2nd order polynomial of this relationship, a and b, respectively, also referred to typically as Forchheimer Coefficients, are in reality, fudge factors, which guarantee a perfect fit between the measured and modelled data. The hydraulic gradient is calculated based upon two additional universal variables which are the fluid density, ρ_f, and the acceleration due to gravity, g. Therefore, the Forchheimer model does not depend on either the value of the particle diameter d_p or the external porosity of the packed column ε_0 but incorporates two additional pegs in the ground not found in any of the fluid models which pertain to the linear (laminar) flow regime. Thus, the solution which the QFFM provides for the N-S equation equivalent, is an orthogonal solution,

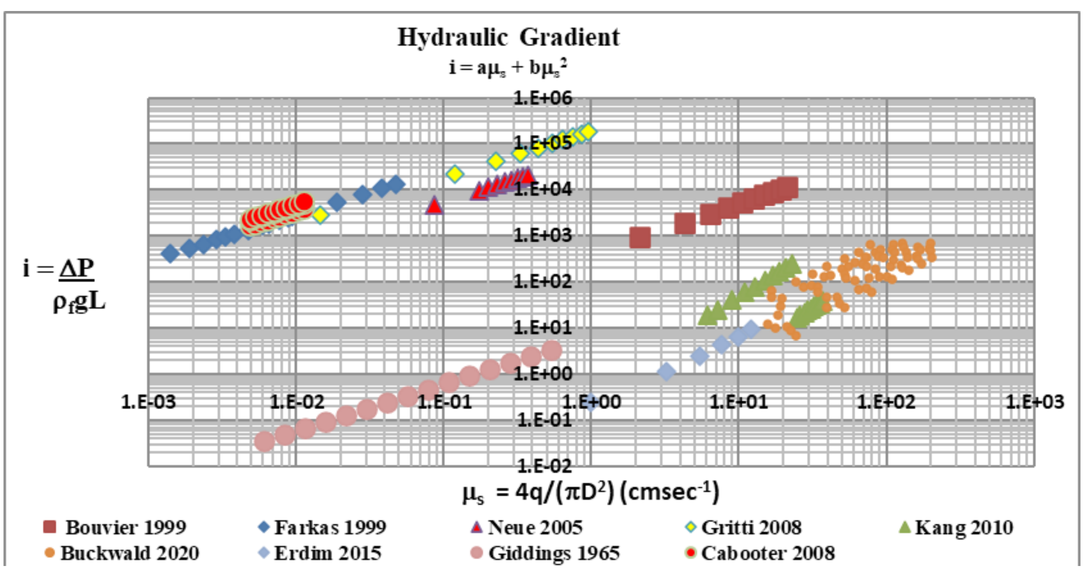


Figure 1: Calculated Permeability for nonporous and partially porous particles.

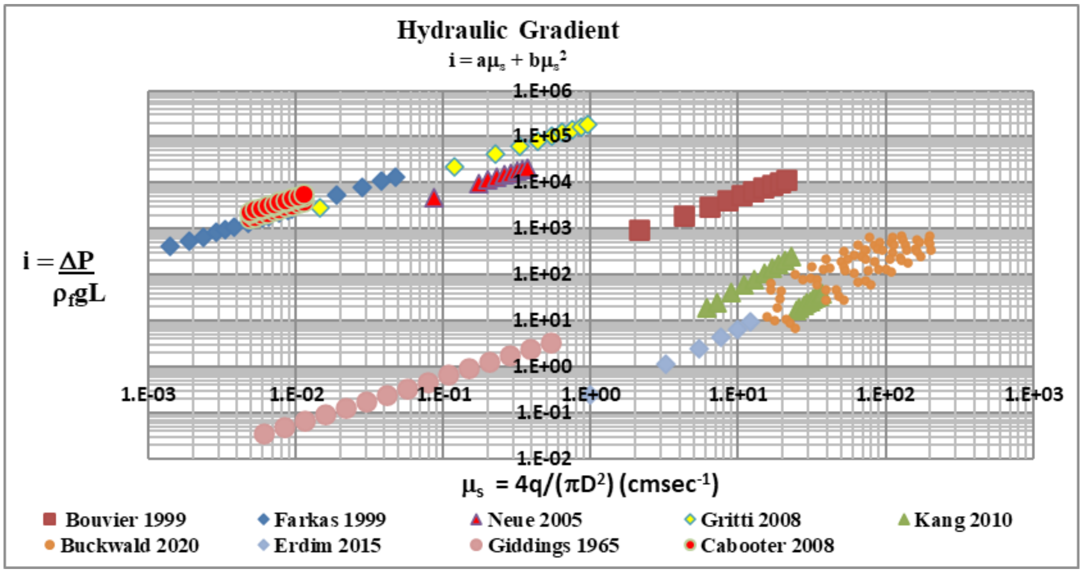


Figure 2: Forchheimer teaching for hydraulic gradient.

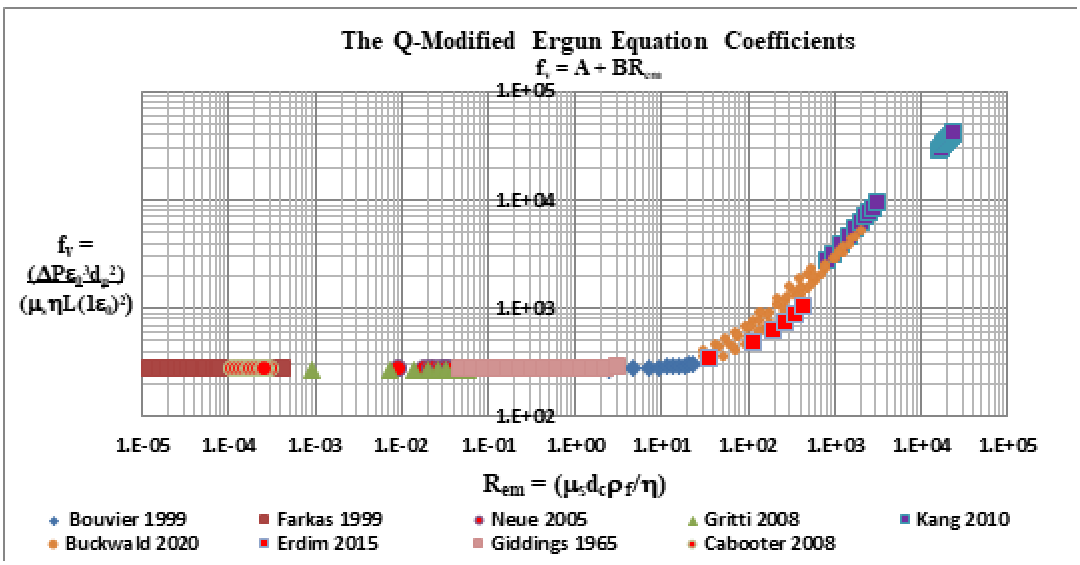


Figure 3: Viscous type friction factor as taught by Ergun.

one axis of which is provided by the combination of variables, fluid density ρ_f and acceleration due to gravity g, and one axis of which is provided by the combination of variables, average spherical diameter equivalent d_p and packed conduit external porosity ε_0.

As shown in Figure 2, hydraulic gradient plots contain axes which are normalized, the y-axis for L and x-axis for D.

Viscous type friction factor f_v

As shown in Figure 3, the datasets are represented as a viscous type friction factor, f_v, versus the modified Reynolds number R_{em}. The parameter $f_v = \Delta P\varepsilon_0^3 d_p^2/(\mu_s \eta L\ (1-\varepsilon_0)^2$, were η is the absolute fluid viscosity. The parameter $R_{em} = \mu_s d_p\ \rho_f / [(1-\varepsilon_0)\eta]$ and the relationship shown in Figure 3 was originally taught by Ergun circa 1951. It enables the calculation of the Ergun coefficients A and B as the intercept and slope of the plotted lines. In the plots herein, of course, the values of A and B represent the coefficients of the Q-modified Ergun model which means that the original Ergun model is modified according to the teaching of the QFFM. Note that in the Q-modified model, the value of A is always a constant = 268.19, but the value of B is not constant and, rather, is defined by the relationship $B = [\lambda/(2\pi\varepsilon_0^3)]$, where λ = the wall normalization coefficient. These values compare to the original Ergun model values of 150 and 1.75 for the values of A and B, respectively.

The Wall-Effect Parameter λ

As taught by the QFFM, the primary wall-effect is due to both the velocity and viscosity of the fluid in the proximity to a confining wall and was identified as the viscous boundary layer by Prandtl circa 1930. In addition, a secondary wall-effect is due to the roughness of the particle surface. The parameter λ in the QFFM quantifies the magnitude of the impact of both these wall-effects on the permeability of any packed or empty column. To isolate the impact of the value of λ, therefore, the QFFM uniquely defines the Dimensionless Permeability parameter Θ in a plot of Θ versus Q_N, where $\Theta = 4Q_N/f_v$, and $Q_N = \delta R_{em}$.

As shown in Figure 4, the 8 datasets for packed conduits containing particles with solid skeletons which have smooth surfaces, are presented. Notice that the range of this plot has the finite value of zero when the value of Q_N is zero, but approaches, asymptotically, the value of $8\pi = 25$ approx. at very large values of Q_N. Note also that all the datasets fall on the line representing $\lambda = 1$ which dictates that packed conduits with smooth particles have no wall effect impact on permeability.

As shown in Figure 5, the packed conduits containing hypothetical particles of free space ($\varepsilon_p = 1$, empty conduits) with smooth conduit walls from the classic Nikuradze study (1933) is presented as well as two reference lines, i.e., λ=1 representing packed conduits with smooth particles, and a line corresponding to the measured smooth-walled Nikuradze data [24]. This latter line has no discrete value for λ assigned to it because its λ value is not constant. This, in turn, is due to the fact that this line represents the primary wall effect, identified as the viscous boundary layer by Prandtl (circa 1933), and has a λ value circa 6.3 at low values of Q_N when the boundary layer is thick but whose λ value decreases as the value of Q_N increases due to the dissipation of the boundary layer. In fact, at extremely large values of Q_N, this line's λ value approaches, asymptotically, the value of unity, corresponding to the complete dissipation of the boundary layer at fully developed turbulence.

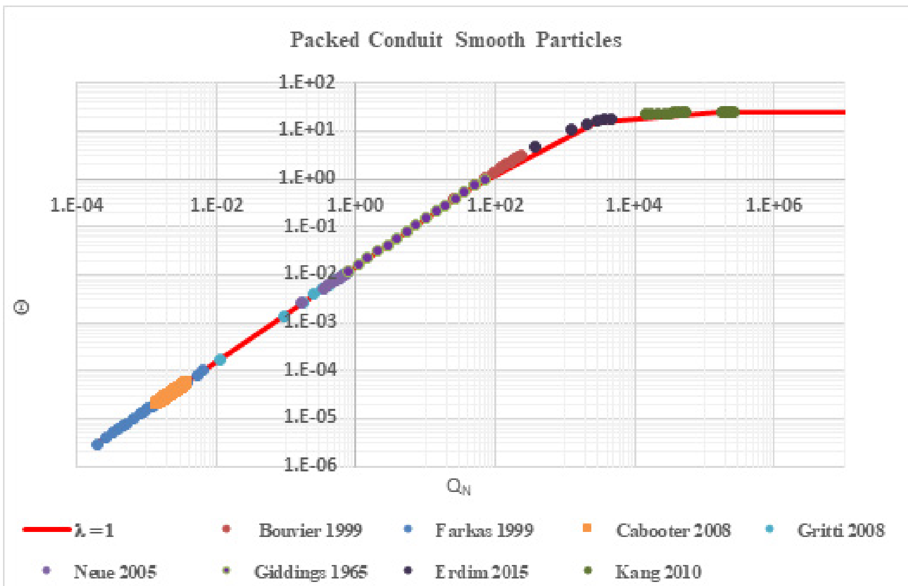


Figure 4: Dimensionless Permeability for smooth particles.

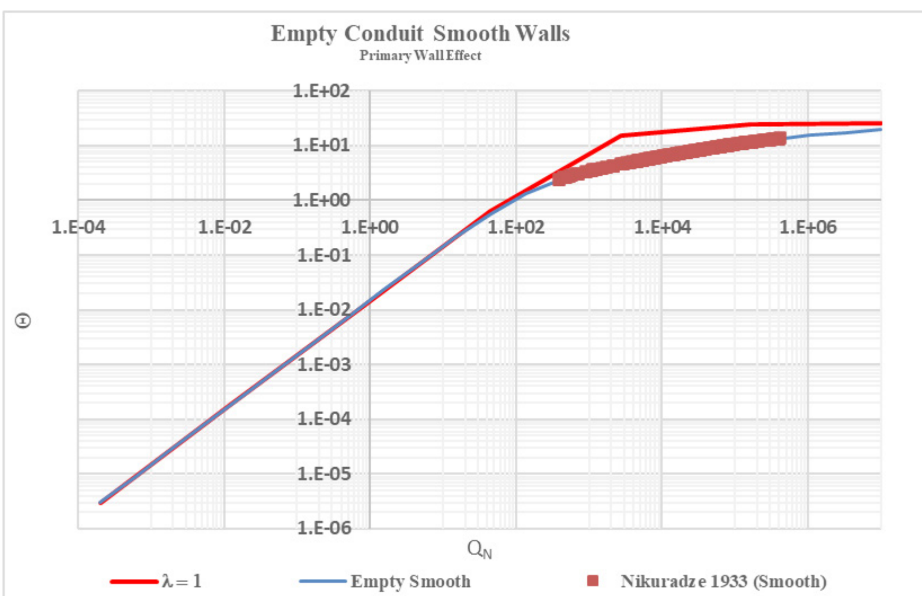


Figure 5: Dimensionless Permeability for empty conduits with smooth walls.

As shown in Figure 6, the packed conduits containing hypothetical particles of free space (ε_p = 1, empty conduits) with roughened conduit walls from the classic Nikuradze study (circa1933) is added [25]. This roughened-wall dataset appears on the righthand side of the line representing the smooth-walled dataset because it is dictated by the impact of the roughened particles punching through the viscous boundary layer. This plot also contains a line representing the value of λ = 15 which dictates that the six levels of wall roughness contained in the Nikuradze study falls in a range between λ = 6.3 approx. and λ = 15 approx.

As shown in Figure 7, the packed conduit containing nonporous solid particles (ε_p = 0) with roughened particle surfaces (0 < k) from the Buckwald study (2020) is added. Note that the rough particle packed conduit dataset of Buckwald falls on the lefthand side of the empty smooth conduit walled line, whereas the empty conduit rough walled dataset falls on the righthand side of the line. This is a very important differentiation and is due to the impact of the tortuosity of the fluid path in the Buckwald data set (particles with solid skeletons) as opposed to the Nikuradze dataset (particles of free space). Thus, it is apparent that the increased tortuosity

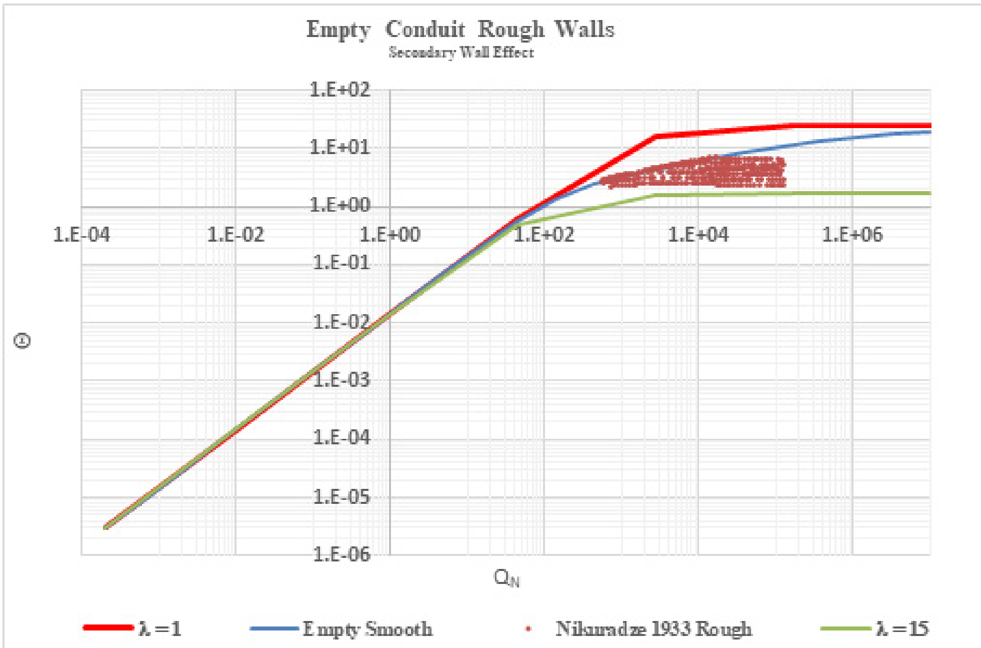


Figure 6: Dimensionless Permeability for empty conduits with roughened walls.

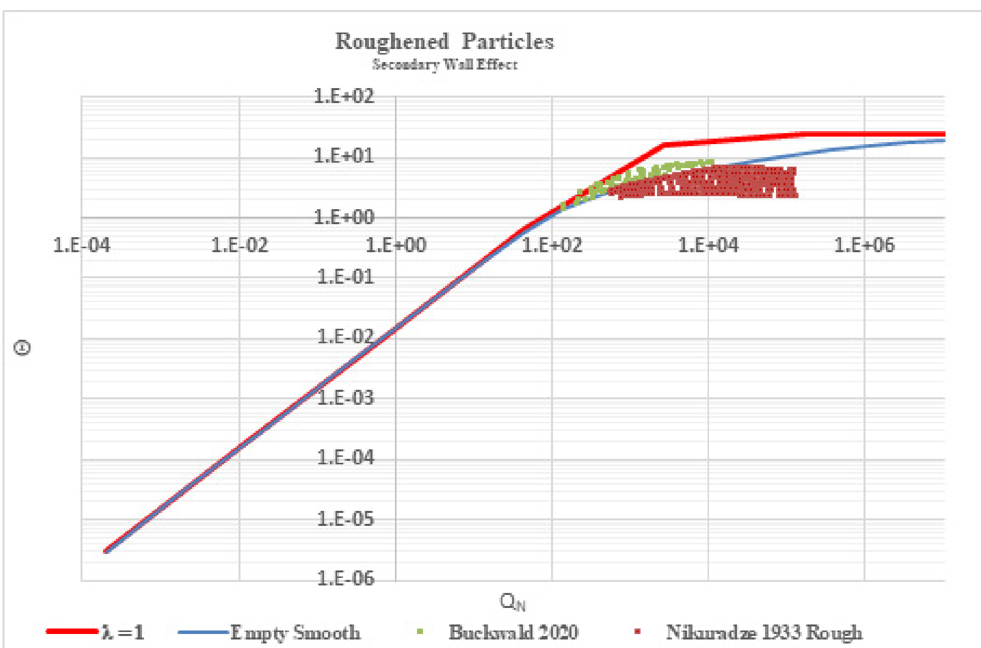


Figure 7: Dimensionless Permeability for solid particles with roughened surfaces.

of a packed conduit containing particles with solid skeletons significantly dissipates the viscous boundary layer which results in a more pronounced impact of roughness, especially at lower values of Q_N.

As shown in Figure 8, the entire landscape of packed conduit Dimensionless Permeability is represented. Note how this plot differentiates between 4 categories of packed and empty conduits, i.e., (1) conduits packed with solid smooth particles, (2) conduits packed with solid roughened particles, (3) smooth-walled conduits packed with hypothetical fully porous particles (empty conduits) and (4) roughened-walled conduits packed with hypothetical fully porous particles (empty conduits).

A Universal Relationship

As shown in Figure 9, the measured datasets for all study samples are shown on a plot of P_Q versus C_Q, which is referred to as Quinn's Law. The parameter $P_Q = (r_h f_v)$, and the parameter $C_Q = \lambda Q_N$, the former represents the normalized pressure gradient also normalized for fluid drag: the latter represents the normalized fluid flow parameter including wall-effect also normalized for fluid drag. As stated above, the parameter $C_Q = \delta\lambda R_{em}$ is uniquely defined in the QFFM, a definition dictated by the Laws of Nature and not recognized heretofore in scientific literature. Note that all measured data fall on the unique straight line defined by this relationship whose intercept and slope represent the values of k_1 and k_2 and which are the universal constants in the pressure flow

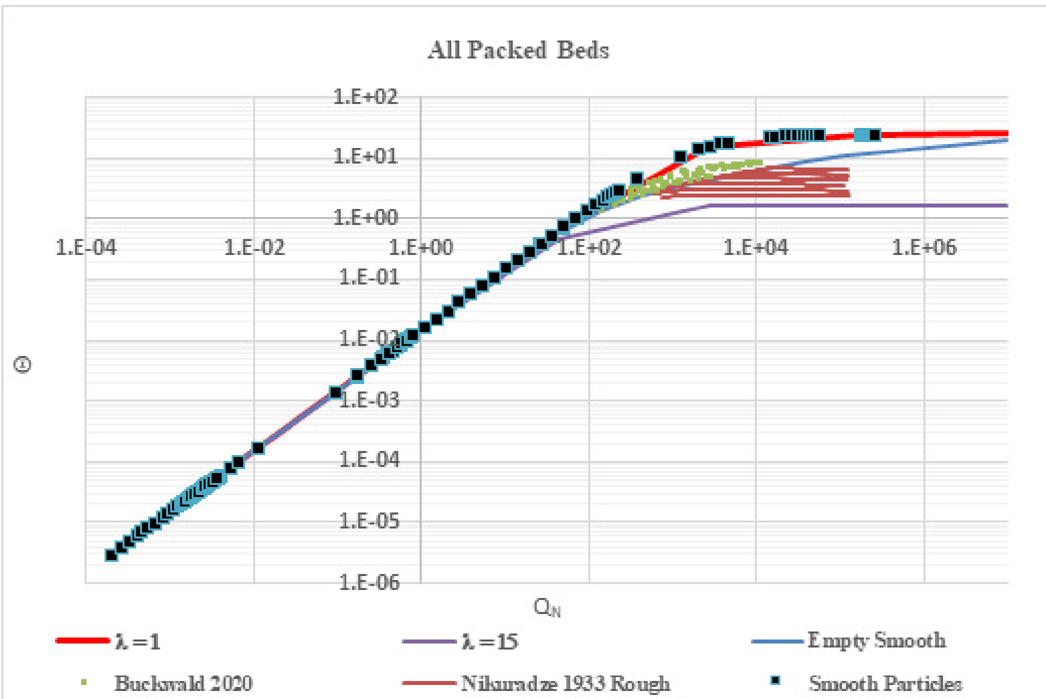


Figure 8: Dimensionless Permeability for all packed beds.

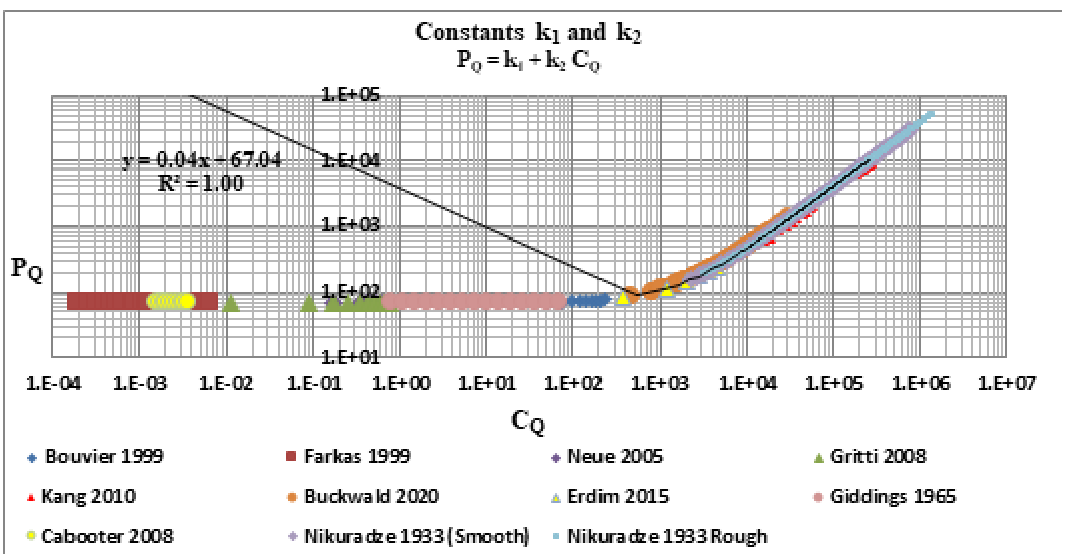


Figure 9: Universal Permeability constants in the pressure/flow relationship.

relationship in closed conduits. The coordinates of this straight line are presented as log-log to provide a landscape view across the entire fluid flow regime.

Data Summary

Supplementary Materials Tables 1A, 1B and 1C contain all the relevant QFFM calculations for the datasets presented.

Conclusions

Based upon analysis of the data sets presented the following conclusions are drawn:

1. The universal constants, $k_1 = 64\pi/3$ (67 approx.) and $k_2 = 1/(8\pi)$ (0.04 approx.) are true constants which do not depend on experimental variables.
2. The value of the Kozeny/Carman constant is really $256\pi/3 = 268.19$ and is also independent of experimental variables.
3. The Dimensionless Permeability parameter Θ defines just 4 categories of packed conduits.
4. The hydrodynamics of all packed conduits is captured by a single mathematical framework.
5. All fluid flow embodiments in closed conduits are hydrodynamically identical at very low values of the modified Reynolds number. This is because, in this region of the fluid flow regime, kinetic contributions to pressure drop are negligible. This, in turn, is a result of the value of the velocity term being less than unity, in this region of the flow regime and, thus, when elevated to the second power, as dictated by the kinetic term, the overall value of the kinetic term is greatly diminished. Consequently, it is only at elevated values of the modified Reynolds number, when the value of the velocity term is greater than unity, and its' value is raised to the second power, thus, greatly enhancing the kinetic contributions, that smooth and roughened surfaces can be differentiated.

1. Chromatographic literature has historically ignored kinetic contributions to packed bed hydrodynamics which has led, in part, to the discrepancies referred to herein. This practice is driven by the fact that chromatographic applications are typically carried out in the laminar flow regime where the hydraulic gradient is a linear function of fluid superficial velocity. The fact that the kinetic term has a negligible impact on bed permeability, in this region of the fluid flow regime, however, does not mean that it can be overlooked, since it serves to anchor the permeability equation in the Laws of Nature, over the entire fluid flow regime.
2. It has been the practice, historically, in Chromatographic circles, to use the measured values for particle diameter, taken outside the packed conduit, in determining the characteristics of packed beds, especially regarding pressure drops. This practice is valid when the particles are rigid but it is not valid when the particles compress under the packing pressures used to pack the column, via the slurry packing methods typically used for small diameter particles. The QFFM methodology, because it is based upon measured values of the packed conduit, identifies the correct value of the spherical particle diameter equivalent d_p existing within the packed conduit, corresponding to the measured values of ΔP and μ_s, even when the particles are in a compressed state. Accordingly, and by extension, it also identifies the correct value of the packed conduit external porosity ε_0.
3. In the Tables of data contained in the supplemental materials, the Waters Corp manufactured partially porous particles, whose trade name goes by Acquity BEH C18, was used in three of our selected examples, Neue et al., Gritti et al. and Cabooter et al. Note that the value listed therein for particle porosity ε_p, varies from the high value of 0.577 to the low value of 0.282. This difference in particle porosity results from the fact that at the enormous packing pressures used for these UHPLC columns, the Acquity BEH C18 polymeric particles are compressing, resulting in lower values for the average spherical particle diameter equivalent, d_p, as well as a reduction in the internal pore volume of the particles.

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