Comparison of porous media permeability: experimental, analytical and numerical methods

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Comparison of Porous Media Permeability: Experimental, Analytical and Numerical Methods

by

Faiz M. Mahdi

Doctoral thesis

Submitted in partial fulfilment of the requirements for the award of the degree of Doctor of Philosophy in Chemical Engineering

Loughborough University

February 2014

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Permeability is an important property of a porous medium and it controls the flow of fluid through the medium. Particle characteristics are known to affect the value of the permeability. However, experimental investigation of the effects of these particle characteristics on the value of permeability is time-consuming while analytical predictions have been reported to overestimate it leading to inefficient design. To overcome these challenges, there is the need for the development of new models that can predict permeability based on input variables and process conditions.

In this research, data from experiments, Computational Fluid Dynamics (CFD) and literature were employed to develop new models using Multivariate Regression (MVR) and Artificial Neural Networks (ANNs). Experimental measurements of permeability were performed using high and low shear separation processes. Particles of talc, calcium carbonate and titanium dioxide (P25) were used in order to study porous media with different particle characteristics and feed concentrations. The effects of particle characteristics and initial stages of filtration as well as the reliability of filtration techniques (constant pressure filtration, CPF and constant rate filtration, CRF) were investigated. CFD simulations were also performed of porous media for different particle characteristics to generate additional data. The regression and ANN models also included permeability data taken from reliable literature sources.

Particle cluster formation was only found in P25 leading to an increase of permeability especially in sedimentation. The constant rate filtration technique was found more suitable for permeability measurement than constant pressure. Analyses of data from the experiments, CFD and correlation showed that Sauter mean diameter (ranging from 0.2 to 168 $\mu$m), the ‘fines ratio’ ($x_{50}/x_{10}$), particle shape (following Heywood’s approach), and voidage of the porous medium (ranging from 98.5 to 37.2%) were the significant parameters for permeability prediction.

Using these four parameters as inputs, performance of models based on linear and nonlinear MVR as well as ANN were investigated together with the existing analytical models (Kozeny-Carman, K-C and Happel-Brenner, H-B). The coefficient of
ABSTRACT

correlation ($R^2$), root mean square error (RMSE) and average absolute error (AAE) were used as performance criteria for the models. The K-C and H-B are two-variable models (Sauter mean diameter and voidage) and two variables ANN and MVR showed better predictive performance. Furthermore, four-variable (Sauter mean diameter, the $x_{50}/x_{10}$, particle shape, and voidage) models developed from the MVR and ANN exhibit excellent performance. The AAE was found with K-C and H-B models to be 35 and 40%, respectively while the results of using ANN2 model reduced the AAE to 14%. The ANN4 model further decreased the AAE to approximately 9% compared to the measured results. The main reason for this reduced error was the addition of a shape coefficient and particle spread (fine ratio) in the ANN4 model. These two parameters are absent in the analytical relations, such as K-C and H-B models. Furthermore, it was found that using the ANN4 (4-5-1) model led to increase in the $R^2$ value from 0.90 to 0.99 and significant decrease in the RMSE value from 0.121 to 0.054.

Finally, the investigations and findings of this work demonstrate that relationships between permeability and the particle characteristics of the porous medium are highly nonlinear and complex. The new models possess the capability to predict the permeability of porous media more accurately owing to the incorporation of additional particle characteristics that are missing in the existing models.

Keywords: Porous Media Permeability, Multivariate Regression (MVR), Artificial Neural Network (ANN), Computational Fluid Dynamics (CFD) and Particle Characteristics
ACKNOWLEDGMENTS

First and foremost, I thank Almighty Allah (God) for blessing me with success and prosperity for the completion of this thesis.

Thanks and gratitude to my parents who have done their utmost to be there for me throughout my life. I would like to express my gratitude to my sponsor, the Libyan Ministry of Higher Education.

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I would like to take this golden opportunity to show my appreciation, gratitude and deep thanks to all those people who helped me in carrying out my research and provided all kinds of support in the Department of Chemical Engineering including the administrative staff: Yasmin, Anna, Ann, Gill and Janey; technical and support staff: Sean, Graham, Paul, Dave, Monika and Kim. I would also like to thank Tony, Jim and Terry for helping me with the fittings and connections.

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F.M. Mahdi;
Feb. 2014
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NOMENCLATURE

- $A$ Area, $m^2$
- $A_p$ Projected Area, $m^2$
- $A_r$ Archimedes number
- $c$ Dry cake mass per unit volume filtrate, $kg \cdot m^{-3}$
- $C$ Solid concentration by volume fraction
- $F_{as}$ Surface shape factor
- $F_{ea}$ Volume shape coefficient when $T=B=L$
- $F_{sa}$ Surface shape coefficient
- $F_{va}$ Volume shape coefficient when $T<B<L$
- $g$ Acceleration due to gravity, $m \cdot s^{-2}$
- $k$ Permeability, $m^2$
- $L$ Bed depth, m
- $L,l$ Length, m
- $\Delta P$ Pressure different or drop, Pa
- $Q$ Volume flow rate, $m^3 \cdot s^{-1}$
- $Q_H$ Heywood Settling Factor
- $R_c$ Cake resistance, $kg \cdot m^{-1} \cdot s^{-2}$
- $Re$ Fluid Reynolds Number
- $Re'$ Particle Reynolds Number
- $R_m$ Filter medium resistance, $m^1$
- $s$ Slurry concentration by mass fraction
- $S_v$ Specific surface area per unit volume, $m^2 \cdot m^{-3}$
- $t$ Time, s
- $u$ Fluid velocity, $m \cdot s^{-1}$
- $U$ Interstitial velocity, Particle Velocity
- $U_o$ Superficial velocity, $m \cdot s^{-1}$
- $V$ Filtrate volume, $m^3$
- $V_p$ Particle Volume, $m^3$
- $x$ Particle Area Diameter, m
- $x_a$ Projected Area Diameter, m
- $x_s$ Surface Area Diameter, m
- $x_{Sv}$ Sauter Mean Diameter, $x_{3,2}$, m
- $x_v$ Volume Diameter, m

- $\varepsilon$ Porosity
- $\mu$ Fluid viscosity, Pa s
- $\rho$ Fluid density, $kg \cdot m^{-3}$
- $\rho_s$ Solid density, $kg \cdot m^{-3}$
- $\rho_b$ Bulk density, $kg \cdot m^{-3}$
- $\alpha$ Specific resistance of filter cake, $m \cdot kg^{-1}$
**ABBREVIATIONS**

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AAE</td>
<td>Average Absolute Error</td>
</tr>
<tr>
<td>ANN</td>
<td>Artificial Neural Networks</td>
</tr>
<tr>
<td>ANN2</td>
<td>Artificial Neural Networks consist of two inputs</td>
</tr>
<tr>
<td>ANN4</td>
<td>Artificial Neural Networks consist of four inputs</td>
</tr>
<tr>
<td>ANFIS</td>
<td>Neuro-Fuzzy Inference System</td>
</tr>
<tr>
<td>CCD</td>
<td>Charge-Coupled Device</td>
</tr>
<tr>
<td>CFD</td>
<td>Computational Fluid Dynamics</td>
</tr>
<tr>
<td>CPF</td>
<td>Constant Pressure Filtration Technique</td>
</tr>
<tr>
<td>CRF</td>
<td>Constant Rate Filtration Technique</td>
</tr>
<tr>
<td>LD</td>
<td>Laser Diffraction</td>
</tr>
<tr>
<td>LASentec FBRM</td>
<td>Focused Beam Reflectance Measurement</td>
</tr>
<tr>
<td>IA</td>
<td>Image Analysis</td>
</tr>
<tr>
<td>NLR2</td>
<td>Simple Nonlinear Regression model consists of two inputs</td>
</tr>
<tr>
<td>NLR4</td>
<td>Simple Nonlinear Regression model consists of four inputs</td>
</tr>
<tr>
<td>FF</td>
<td>Feed-Forward function</td>
</tr>
<tr>
<td>FFP</td>
<td>Feed-Forward Back-Propagation</td>
</tr>
<tr>
<td>IBP</td>
<td>Improved Back Propagation</td>
</tr>
<tr>
<td>H-B</td>
<td>Happel-Brenner Model</td>
</tr>
<tr>
<td>K-C</td>
<td>Kozeny-Carman Model</td>
</tr>
<tr>
<td>RMSE</td>
<td>Root Mean Squared Error</td>
</tr>
<tr>
<td>R²</td>
<td>Coefficient of Correlation</td>
</tr>
<tr>
<td>MVR</td>
<td>Multivariate Regression</td>
</tr>
<tr>
<td>MNN</td>
<td>Modular Neural Network</td>
</tr>
<tr>
<td>MLP</td>
<td>Multilayer Perceptron</td>
</tr>
<tr>
<td>PCA</td>
<td>Principal Component Analysis</td>
</tr>
<tr>
<td>PCR</td>
<td>Principal Component Regression</td>
</tr>
<tr>
<td>PLSR</td>
<td>Partial Least Squares Regression</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
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</table>
CHAPTER ONE

INTRODUCTION

1.1 Background

The permeability of solids forming a porous medium is an important parameter that determines the frictional loss during fluid flow through that medium. Permeability can be used to predict the flow rate of fluid through the material for a given pressure difference when modelling the behaviour of fluid transport in any porous medium, or packings. In many filtration experiments in the laboratory, the objective is to determine the filter cake permeability or, alternatively, specific cake resistance to filtration and how it varies with pressure as well as information on the filter medium resistance after it has stabilised (Tarabara et al. 2002, Tiller 1953). Mauran et al. (2001) argues that permeability of the porous media is essential to establish the relationship between the fluid flow rate and pressure gradient for application of Darcy’s law. In this law, the permeability is a constant that defines the rate at which a viscous fluid flows through a porous medium (Berryman et al. 1987, Collins 1961, Muskat et al. 1946). Permeability is a unique property of a porous medium and, for incompressible materials, is independent of the absolute pressures and velocities within the flow system as well as the nature of the fluid (Wyckoff et al. 1933).

According to Darcy’s law the volumetric flow rate ($Q$) of liquid through a porous material is proportional to the pressure difference ($\Delta P$) across the material, inversely proportional to the length ($L$) of the packing and proportional to the cross sectional area ($A_c$) (Hall et al. 2002). The SI unit for permeability is $m^2$ but it is more appropriate in the oil industry to state permeability in Darcys [D] or milliDarcys [mD]. Darcy’s law is often applied to a single-phase fluid flow in a porous medium when the flow is laminar (Loosveldt et al. 2002). The permeability of a liquid such as water, through a porous sample can be measured simply by allowing it to flow continuously through the sample with a constant pressure gradient (Loosveldt et al. 2002). The volume of liquid that flows through the sample is measured at specific time intervals, which can be used to calculate the volumetric flow rate (Despois et al. 2005, Baraka-Lokmane 2002).
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The porous medium discussed above refers to a body of particles with certain characteristics. Even for the same material or within a given sample, the particles may exhibit variation in characteristics. They are usually irregular in shape, different in size, vary in size distributions and have different surface morphology. These characteristics affect the industrial processes in different ways and their effects make particle characterisation an important application in many manufacturing processes. Generally, in industry, particle shape and size are the most important factors, which affect particulate system behaviour such as flow and reaction properties (Rhodes 2008, Wijngaarden et al. 1998). In particular, particle characteristics such as particle size, particle size distribution (especially fine particles), solids concentration, particle shape and orientation influence the permeability of a porous medium (Fu et al. 2012, Biggs 2006).

There are a number of well-known analytical expressions for the prediction of permeability e.g. Kozeny-Carman (K-C) and Happel-Brenner (H-B) but their reliability is limited (Mahdi 2008, Carman 1956, Brinkman 1949). The K-C and H-B models assume that the particles are in fixed geometry, rigid and are in point contact with each other (Rushton et al. 2000). It has been reported that both models work best within a voidage range of $0.4 < \varepsilon < 0.7$ (Davies et al. 1980). Also, the K-C model and many other models contain ‘coefficients’ whose values often depend on the process and material properties and as such there exist no relationships for their predictions (Tien et al. 2013).

The determination of permeability through experiments is time consuming and is not very cost effective when performing a large scale filtration work. Meanwhile, the available models for its prediction are limited in applications based on the factors listed above. To overcome these challenges, there is a need to develop new models that allow the prediction of permeability using different variables under various process conditions. This is the motivation of this work; it aims to establish a new model that can predict permeability at very low and high concentrations, and also for different sizes and shapes of particles.

In this project, the particle permeabilities will be obtained from three sources: literature, Computational Fluid Dynamics (CFD) and lab experiments. The experiments will involve different processes including permeation, filtration and
INTRODUCTION

sedimentation. Furthermore, different kinds of materials will be used in this research in order to further quantify the relationship between porous media permeability and particle characteristics.

Based on the results from the above sources, the new models will be developed using two different numerical methods: Multivariate Regression (MVR) and Artificial Neural Network (ANN). In MVR, principal component regression (PCR), partial least squares regression (PLSR) and simple nonlinear regression (NLR) will be used. All of these methods are common predictive models building techniques (Saleemi 2011). The ANN model will be designed, trained and validated using a large number of data sets, which will be obtained from laboratory experimental work, CFD models and the literature. This step will continue until an acceptable model performance is reached. The advantage of using ANN is due to its ability to approximate arbitrary continuous functions that are based on a set of data supplied to its network to predict particle permeability.

1.2 The research aims and objectives

The aims and objectives of this research include the following:

- To achieve an enhanced understanding of porous media permeability under different conditions.
- To obtain reliable data of permeabilities for porous media from previous studies for several materials under various process conditions.
- To measure the permeabilities of porous media experimentally for a number of different materials under different conditions.
- To identify the significant parameters that have the highest effect on the permeability values both experimentally and numerically.
- To develop a new approach to predict the permeability of porous media better than existing models using Multivariate Regression (MVR) and Artificial Neural Network (ANN).
- To provide a numerical model with increased accuracy when predicting porous media permeability.
- Finally, to compare all of these different model results to the measured permeability in order to find the model with the best fit and lowest error ratio.
1.3 Research Methodology

In this study, various approaches and techniques were used. The data were obtained from diverse sources (experimental, CFD simulations and literature). Pre-processing of the data, investigation of the design and evaluation of numerical models were performed in order to achieve the ANN and MVR models with excellent performance. Finally, the predicted results from analytical (existing models) and numerical models (developed in this work) were examined in the light of the measured results with the aim of establishing a more reliable model.

1.4 Research Contributions

The main contributions of the work presented in this thesis can be summarised as follows:

- The provision of a comprehensive but concise literature review on particle characterisation methods for analysing shape and size of particles. Also, experimental and numerical methods for calculating the permeability of solid-liquid porous media were shown. This includes different permeability prediction models and their limitations.
- The aims of this study were achieved by obtaining permeability data (from literature, CFD and experimental) and using ANN and MVR approaches to develop a numerical model.
- An appropriate method of categorisation for measuring particle shape coefficient using Heywood’s technique is simplified and used.
- Cluster formation and its effect on the permeability of porous media within different solid-liquid separation systems including sedimentation, permeation and filtration were investigated.
- In order to improve the knowledge of filtration equipment design, the effect of both filtration techniques (constant pressure and constant rate) on the specific cake resistance measurement was studied and the constant rate technique was found and shown to be more reliable for measuring the permeability of porous media.
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- A new simple and clean lab system for constant rate filtration (CRF) technique was designed, calibrated, used and presented in this work.
- The changes during the initial stage of filtration on the porous media permeability measurement was reported. However, it was shown that, the initial stage phenomenon does not have any long-term significant influence on the average porous media permeability.
- An incremental and iterative method was developed to calculate the specific cake resistance by measuring the total pressure difference applied across a filter medium.
- Four parameters (size, concentration, shape and size distribution of particles) were found numerically (PCA and CFD) and experimentally to have the most significant influence on the prediction of permeability.
- A systematic model development approach for permeability determination in porous media using four inputs is presented. A novel equation was developed to predict the permeability of porous media using the nonlinear regression approach; this model can be simply applied in Excel.
- The methodology for training the ANN MATLAB code was shown. This methodology is vital to the establishment of an ANN network that accurately learns the nonlinear relationships between the independent and the dependent variables.
- A CFD model was designed and validated for further use in porous media permeability investigation and prediction.

1.5 Thesis Structure

The thesis comprises of seven chapters which are presented in the following order:

Chapter 1 introduces the definition of the problem, the significance and the background of permeability and particle characteristics and their importance in industrial processes. The experiments for permeability measurement and well-known analytical models for predicting permeability with their limitations were discussed.

Chapter 2 reviews different types of particle characterisation for size, shape, packing and porosity of particle beds. In this chapter, the theoretical background of permeability models in different processes is explained with their limitations. Also, it
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describes the computing methods, e.g. Computational Fluid Dynamics (CFD) Multivariate Regression (MVR) and Artificial Neural Network (ANN) in MATLAB. Design steps and different kind of ANN functions are reported and discussed together with their advantages, disadvantages and applications.

Chapter 3 explains different kinds of particle characterisations by using various methods: Scanning Electron Microscope (SEM), Laser Diffraction (LD) and Image Analysis (IA). Also, compressibility, Zeta potential, cluster formation and shape coefficient of all solids were investigated and tabulated under various conditions.

Chapter 4 presents experiments and simulation set-up and methodology. Different methods of solid-liquid separation were used. An appropriate method of shape coefficient ($F_{\text{w}}$) measurement explained in detail. Furthermore, a method was explained to calculate the specific cake resistance ($\alpha$). An investigation on the ANN model design and input variables (numerically) was made. With regard to the computer simulation work, three different cell models were designed and validated for different conditions.

Chapter 5 presents experimental investigations on the four input variables (particle size, concentration, shape and size distribution). A study on the filtration techniques and benefits as well as specific resistance and permeability were shown. Also, initial stages of filtration and cake compressibility are examined at different conditions.

Chapter 6 explains three different sources where permeability data were obtained. These include literature, experiments, and simulation models. Furthermore, a numerical investigation of the input variables using the CFD model was shown.

Chapter 7 presents the results of the numerical models (ANN and MVR) as well as the analytical models (K-C and H-B). Design steps and different kinds of functions are reported and discussed together with model validation.
CHAPTER TWO

LITERATURE REVIEW

2.1 Particle Characterisation

Particle characterisation is one of the most important areas in all aspects of particle technology, handling and separation processes such as filtration and sedimentation. It is a primary task required in a process involving solid particles that does not only involve their intrinsic parameters i.e. size, density, shape, and morphology, but also their dynamic behaviour such as drag coefficient and terminal velocity. Particles are usually irregular in shape and have different surface morphology, because of this the measurement of particle characteristics (size, shape and surface morphology) require complete analysis. Moreover, the results may not be completely consistent depending on the method applied. Although, some methods may be more appropriate than others for certain selected applications (Yang 2003).

According to Kaye (1999) and Fayed et al. (1997) there are several different ways to graphically summarize and describe the size and shape information of a particle based on microscopic analysis of the particle, i.e. classical size distribution histogram, chunkiness distribution and the chunkiness-size domain (Kaye method). In any process where a fluid flows through a porous medium such as sedimentation, permeation and filtration, the permeability of the solids forming the porous medium governs the frictional loss. Hence, the observed rates of filtration, permeation and sedimentation are determined by that permeability. Also, a reliable permeability predicting method based on the particle size distribution would facilitate the process design and facilitate better understanding. This remains an area of interest for researchers for many years. The nature of the solids and how their particle size analysis is determined is also of great importance when dealing with fine sized particles, as they often form clusters containing many primary particles, and thus influencing the predicted permeability. The best-known factors influencing the permeability of a porous medium are: particle size, particle size distribution (especially fines), solids concentration, particle shape and orientation (Biggs 2006).
2.1.1 Particle Size

There is a wealth of published literature investigating the influence of particle morphology, particle size distribution, pH and nature of particle-particle interactions on filtration (Wu et al. 2003, Intani et al. 1997, Wakeman et al. 1991). In all cases, the particle size distribution within a porous medium is critically important for determining the permeability and the rate of filtration. However, it is important to note that the size (or size distribution) of the particle, or cluster of particles, is of particular importance. Particle size measurement is a favourite subject of study for powder technologists and many researchers (Hawkins 1993).

Particle size affects a number of material characteristics. Regular shaped particles that are not spherical can usually be characterised by two or three dimensions. For example, a cubic particle can be uniquely defined by a single dimension, while cuboids require all three dimensions, which are length, width and height. In some cases only two dimensions are required for regular isotropic particles such as cylinders, spheroids and cones. On the other hand, irregular particles of practical interest cannot be uniquely defined directly, but their sizes are usually defined based on certain reference properties. The choice of any particular diameter for characterisation of an irregular particle depends on the intended application in most cases (Salman et al. 2007, Yang 2003). When the particle is non-spherical the determination of the single particle size is not a simple task. Either particle orientation or measuring direction affects the measurement of both common statistical diameters (Feret and Martin), (Masuda et al. 2007).

Rhodes (2008) reported a number of different geometrical sizes as well as different dynamic ones together with the statistical diameters, which include Martin, Feret, Image sheared and Equivalent circle diameter as shown in Figure 2.1. Particle samples have a very wide range of size and shape for the same material, which is not easy to determine. Resolving this quantitatively has posed a problem for a long time and is still a matter of concern for particle technologists. Microscopic analysis and counting the particles is one of the significant characterisation techniques used in particle technology. It is slow and prone to operator bias when performed manually. In recent years, a modern technique has been developed, such as the use of a microscope attached to a camera and computer. This has been an important tool.
in particle characterisation and essential for assessing the material quickly (Holdich 2002). In such cases, particle shape should be considered since microscopical measurement does not generally determine the thickness of the particle. Moreover, microscope diameter and subsequently projected area that being the equivalent spherical area will have the same projected area as the particle when viewed in a direction perpendicular to the plane of greatest stability (Holdich 2002, Allen 1997).

Masuda et al. (2007) found that, the particle size distribution has been usefully defined as the state of subdivision of a material. For a smooth dense sphere, size ($x$) is equivalent to diameter ($D_p$), although in most industrial applications, size ($x$) is equivalent to diameter ($D$) only through the use of an adjustment factor ($F$) where $D_p = Fx$ and $F$ can be a shape factor or a combined shape and extinction coefficient in the case of optical methods. Without knowledge of $F$ the accurate size measurement of an irregular particle is difficult. There are many common methods of expressing the size of irregular particles using dimensions of particles (Merkus 2009, Masuda et al. 2007, Yang 2003, Allen 2003, Holdich 2002, Clift et al. 1978).

Microscopy and image analysis (IA) are used with many methods to obtain the size of particle. However, the sieve diameter ($x_s$), the volume diameter ($x_v$), the surface diameter ($x_i$) and the surface volume diameter ($x_{sv}$) are of general interest for applications in packed beds and fluidized beds (Masuda et al. 2007, Clift et al. 1978). The surface volume diameter ($x_{sv}$) is the most relevant diameter in many particle processes. According to Merkus (2009) there is a relationship between the mean Feret diameter ($x_F$) and the particle perimeter ($x_c$) when all possible orientations of the particle projection are taken into account that is equal to $x_F = x_c / \pi$. Similarly, another relationship between mean projected area in different particle orientations ($A_w$) and the particle surface area ($S$) which is $A_w = S / 4$. 

![Image of Statistical Diameter used in Image Analysis. Source: Holdich (2002)](image-url)
2.1.2 Particle Shape

Gabitto et al. (2008) concluded that, particles have a wide range of shapes from roughly spherical pollen and fly ash to cylindrical asbestos fibres and irregular mineral particles. The fact is that many effects on the measurement of particle parameters and the change in size of particles and its distribution can be related to changes in the shape of particles. Also, shape controls the behaviour of the particles in different kinds of process industry (Hawkins 1993). Although, the particle shape is very important, it is normally ignored and that is why only a limited amount of research has been done and published (Salman et al. 2007, Hawkins 1993, Nikolakakis et al. 1985). The difficulty of sorting fine powder into different shape fractions, especially for particle size less than 80 μm in diameter, is an important reason behind the limitation on shape research (Salman et al. 2007, Nikolakakis et al. 1985).

Endo et al. (2002) found that particle shape has an effect on the estimation of permeation resistance based on the channel and drag theory. In general, a model based on the drag theory is more realistic in the experimental comparison and practical applications than one based on channel theory. This is due to the dynamic shape factor in a drag model being a constant, while both surface and volume shape factor in a channel model are dependent on the selection of particle diameter. Chin et al. (1986) reported that some work has been done on irregular shaped particles. Particle shape can be defined by Scanning Electron Microscopy (SEM) and Image Analysis (IA) in 2 dimensions, and then documented as a micrograph. Also, it can be quantified by using IA in conjunction with SEM. To describe a 3D particle is usually more difficult and complex than it appears at the start. For simplicity it is convenient to describe particle size as one single number. Some examples of particle shapes and its definition were reported (Merkus 2009).

Holdich (2002) stated that, shape of a particle is an important factor in particle size and permeability measurement, which is a fundamental piece of information for a large number of industries including Pharmaceutical, Chemical, Petrochemical, Food, Cement and Building Materials. Furthermore, it is important in many other particle processes such as deposition and packing, porosity and fluid flow (Merkus 2009). There are many different methods that might be used to measure the shape of
particle and its distribution e.g. Triangular grid graph paper, the method of selecting and sizing particles by using a grid (Reticule), R-theta and the fractal dimension of rugged profile methods. In addition, one of the disadvantages of using these methods is the variations in the reproducibility (Merkus 2009, Santamarina et al. 2004, Holdich 2002, Kaye 1999, Fayed et al. 1997).

Allen (2003) and Barrett (1980) investigated the relationships between various aspects of shape and tried to find the most effective parameters to estimate analytically. They found that, there are many parameters for describing the shape of a particle but none of them is universally accepted. Confusion exists about which parameter needs to be measured and how they are related. An alternative approach to the description of particle shape uses the ratio based on elongation (particle length to width) and flakiness (particle width to thickness), (Merkus 2009, Allen 2003).

According to Jentys (1999) there are many different methods and types of equipment, which are used to determine the shape and geometry of particles. Generally, the Lasentec (FBRM), Malvern and Coulter are first calibrated against near mono-sized polystyrene latex particles of known size (Holdich 2002). Using the Laser diffraction measurement some methods have been able to evaluate the average aspect ratio (Merkus 2009). Particle behaviour is affected by the three principal scales in particle shape including sphericity, angularity and roughness. Each scale reflects aspects of the formation history, and participates in determining the global behaviour of the particle material mass, from particle packing to mechanical response (Santamarina et al. 2004).

A low cost CCD camera and monitor interfaced to a computer with commercially available software programs for image analysis has been widely used instead of many of these methods (Kaye 1999, Masuda et al. 2007). Masuda et al. (2007) reported that new information technology and advanced computer image processing technology has progressed making it easy to obtain image information and geometrical features of particles. From literature reviews, it is found that, various quantitative methods for expressing particle shape have been proposed and adopted. The description is sometimes suitable to express irregular shape and makes it easy to understand the shape visually. Either, two or three dimensional (2D,
3D) geometrical properties might be used to calculate quantitative shape and can be calculated by comparing with physical properties of the reference shape.

### 2.1.2.1 Shape factors

Shape factors are one of the most widely used methods to describe the shape of particles (Clift et al. 1978). The equivalent sphere is the sphere with the same value of one of parameters: e.g. volume \( V_p \), surface area \( A \), projected area \( A_p \) or projected perimeter \( P_p \). Although, the particle shape factor is the ratio of a characteristic parameter of the above parameters to the corresponding value for the equivalent sphere as described in Eq. (2.1.1).

\[
F_{va} = \frac{V}{x_a} \quad \text{where} \quad x_a = \sqrt[4]{\frac{4A_p}{\pi}} \quad (2.1.1)
\]

In addition, the Wadell method (Allen 2003, Clift et al. 1978) is one way to determine the particle projected diameter \( x_a \) without knowing the \( A_p \) as in Eq. (2.1.2).

\[
\psi = \frac{A_v}{A} \quad (2.1.2)
\]

where, \( A_v \) is the surface of a sphere having the same volume as the particle, and \( A \) is the actual surface area of the particle. From Eq. (2.1.2) it can be found that the sphericity \( \psi \) of a true sphere is equal to unity, which means that the more the aspect ratio departs from unity the lower is the \( \psi \), however, it is not easy to determine \( \psi \) directly for irregular particles (Freireich et al. 2011, Gabitto et al. 2008, Clift et al. 1978). Also, Wadell (Allen 2003, Clift et al. 1978) studied the degree of circularity \( \phi \) and established its relationship with the perimeter of a sphere with equivalent projected area \( P_a \) and the projected perimeter of the particle \( P_p \) as shown in Eq. (2.1.3).

\[
\phi = \frac{P_a}{P_p} = \frac{\pi x_a}{P_p} \quad (2.1.3)
\]

Unlike sphericity \( \psi \), circularity \( \phi \) can be determined from microscopic or photographic observation. Even though \( \phi \) is purely used empirically, it allows the correlation of flow dependence on particle orientation (Allen 2003). Although, \( \phi \) can be very useful for some applications, it is not suitable for all situations (Allen 2003, Clift et al. 1978). Freireich et al. (2011), Gabitto et al. (2008) and Alger et al. (1968)
discussed the limitations of such shape factors and found that, to date, there is no definition of a universal shape parameter that will work in every case. In reality, careful consideration is necessary to determine the most suitable parameter for each specific application.

From the above information, it can be seen that, there is no a universal shape parameter that can be used to define particle shape. As a result, shape description and methods to simplify the description of particle shape were considered in more detail and that due to Heywood (Heywood 1971; Heywood 1962) was given particular attention.

2.1.2.2 Shape Coefficients

According to Allen (2003), Eriksson et al. (1997), Freshwater (1987) and Clift et al. (1978), Heywood’s solution is based on two steps. First, to understand the generic properties of the particle and then to distinguish it from other shapes. Heywood (Heywood 1971; Heywood 1962) postulated that, surface and volume are the two most important properties of particles. As displayed in Eq. (2.1.4) the surface area (S) is:

\[ S = \pi x_s^2 = F_{sa} x_a^2 \]  

(2.1.4)

where, \( x_s \) is the equivalent spherical diameter by surface area of the particle, \( x_a \) is the projected area diameter measured by microscopy and \( F_{sa} \) is the surface shape coefficient, which can be found using Eq. (2.1.5).

\[ F_{sa} = \frac{\pi x_s^2}{x_a^2} \]  

(2.1.5)

Also, volume shape coefficient \( (F_{vu}) \) can be obtained using particle volume \( (V_p) \) as in Eq. (2.1.7).

\[ V_p = F_{vu} x_a^3 \]  

=> \[ \frac{\pi}{6} x_v^3 = F_{vu} x_a^3 \]  

(2.1.6)

Rearranging Eq. (2.1.6) will give \( F_{vu} \):

\[ F_{vu} = \frac{\pi x_v^3}{6 x_a^3} \]  

(2.1.7)
where, \( x_v \) is the equivalent spherical diameter by volume. Regular shape coefficients can easily be calculated using these correlations.

### 2.1.2.3 Heywood’s Equation Derivation Steps

Normally, particles have different types of shapes that could be elongated and/or flattened (Allen 2003). The thickness of a particle cannot be measured in general by using a microscopic picture where the projected area diameter is the significant parameter found by this method. It is necessary to mention that the characteristic diameter used to represent the particle size affects the numerical value of the volume coefficient. Heywood (1971) used projected area diameter \((x_a)\) to calculate the volume coefficient. One important reason to use \(x_a\), is that, it can be determined for all sizes of particle types using microscopic pictures (Heywood 1971). By considering a particle resting on a plane, Heywood (1962) investigated the degree of distortion of particles by defining its dimensions in three orthogonal directions. The thickness \((T)\) is perpendicular to the plane of greatest stability, the breadth \((B)\) and the length \((L)\) being parallel to this plane, so that \(T < B < L\), as shown in Figure 2.2 (Heywood 1962).

![Figure 2.2: Heywood’s Dimensions](image)

**Figure 2.2: Heywood’s Dimensions**

Sources: Fayed et al. (1997), Allen (2003), Freshwater (1987)

According to Heywood (1963) the particle geometric shape and particle proportions need to be distinguished by taking into account shape definition problems. The proportions are defined by two ratios that are \( n \) and \( m \) where \( n \) is the ratio of the length to the breadth (elongation ratio) and \( m \) is the ratio of the breadth to the thickness (flatness ratio) as shown in Eqs. (2.1.8), (2.1.9) and (2.1.10). There are two other important ratios that could be used to define shape. The first one is the ratio of particle projected area to the area of the circumscribing rectangle and it is called the area ratio \((r_a)\). The second ratio is the ratio of the mean thickness of the particle to the maximum thickness that is called the prismoidal ratio \((P_r)\).
Elongation ratio, \( n = e_2 = L/B, \quad B = L/n, \quad L = Bn \) \hspace{1cm} (2.1.8)

Flatness ratio, \( m = e_1 = B/T, \quad B = mT, \quad T = B/m \) \hspace{1cm} (2.1.9)

\[ B = L/n = mT, \quad L/T = mn \] \hspace{1cm} (2.1.10)

From Eqs. (2.1.8) and (2.1.9) the surface and volume shape factors can be combined. The projected area of a particle that is circumscribed by a rectangular parallelepiped (see Figure 2.2) \( L, B, T \) can be calculated using Eq. (2.1.11).

\[
\text{projected area} = \frac{\pi}{4} x_a^2 = r_a BL = r_a nB^2
\] \hspace{1cm} (2.1.11)

where, \( r_a \) is the area ratio \( r_a = \frac{\pi}{4} \frac{x_a^2}{BL} \) that will be equal to \( r_a = \frac{\pi}{4} \) in the case of a sphere or cubic particles. Similarly, the volume will be equal to the projected area \( (A_p) \) times the mean thickness, which is equal to \( P_r \) times \( T \), where \( P_r \) is the prismoidal ratio \( (P_r = \text{mean} T/ \text{max} T) \). The prismoidal ratio depends upon the shape of particle, which is \( P_r = 1 \) in case of cube and plate particle where the mean \( T \) is equal to \( \text{max} T \). Further, \( P_r < 1 \) when the particle is almost a sphere or prism and it is \( P_r << 1 \) for needle and flake particles. The Heywood shape coefficient \( (F_{va}) \) is an empirical parameter based on the projected area \( (A_p) \) and profile of particle.

\[ F_{va} x_a^3 = r_a LB P_r T \] \hspace{1cm} (2.1.12)

Using information from Eq. (2.1.8) and (2.1.9), Eq. (2.1.12) will be equal to

\[ F_{va} x_a^3 = r_a nB^2 P_r \frac{B}{m} \] \hspace{1cm} (2.1.13)

From the area ratio equation the projected area diameter will be as shown in Eq. (2.1.14)

\[ x_a = \left( \frac{4}{\pi} BL r_a \right)^{1/2} \] \hspace{1cm} (2.1.14)

By combining Eqs. (2.1.13) and (2.1.14):
After rearranging Eq (2.1.15) the $F_{va}$ can be found as in Eq. (2.1.16).

$$F_{va} = \left( \frac{\pi}{4} \right)^{\frac{3}{2}} \frac{n(B)^{\frac{3}{2}} P_r}{m(r_a)^{\frac{3}{2}} (L)^{\frac{3}{2}}}$$

(2.1.16)

where $B/L = 1/n$

$$F_{va} = \left( \frac{\pi}{4} \right)^{\frac{3}{2}} \frac{nP_r}{m(r_a)^{\frac{3}{2}} (n)^{\frac{3}{2}}}$$

(2.1.17)

Finally, from Eq. (2.1.17) the volume coefficient equation will be as shown in Eq. (2.1.18).

$$F_{va} = \frac{\pi (\pi)^{\frac{3}{2}} P_r}{8 m(r_a)^{\frac{3}{2}} (n)^{\frac{3}{2}}}$$

(2.1.18)

The value of $F_{va}$ is $\pi/6 = 0.524$ for a spherical particle and the averages are in the range of $0.25 – 0.2$ for most minerals and less than 0.1 for a very flat particle. Using the valid equation (Eq. (2.1.18)) together with information from Table 9.1 in Appendix-A the volume shape coefficient can be calculated and from direct observation it is possible to determine into which class the particle fits. Under isometric condition, where $L=B=T$ for $n=m=1$, Eq. (2.1.18) will be rearranged to be Eq. (2.1.19).

$$F_{ea} = \frac{\pi (\pi)^{\frac{3}{2}} P_r}{8 (r_a)^{\frac{3}{2}}}$$

(2.1.19)

where, $F_{ea}$ is the volume coefficient of shape under isometric condition, $P_r$ is the prismoidal ratio, $r_a$ is the area ratio and $m$ is the flatness ratio. The relationship between $F_{va}$ and $F_{ea}$ is shown in Eq. (2.1.20).

$$F_{av} = \frac{F_{ea}}{m\sqrt{n}}$$

(2.1.20)

The value of $F_{va}$ for regular and irregular particle shapes can be estimated using Eq. (2.1.19) and Table 9.1 in Appendix-A (Clift et al. 1978). Furthermore, evaluating the
number, mean size, weight and density of closely graded fractions may be used to find the value of \( n \). Heywood (1971) solved the problem of the surface shape parameter determination by using the idea of geometric similarity. The surface shape factor \( (F_{as}) \) is found using the empirical relationship as in Eq (2.1.21).

\[
F_{as} = 1.57 + C \left( \frac{F_{as}}{m} \right)^{4/3} \frac{n + 1}{n} \tag{2.1.21}
\]

where, \( C \) is a constant that depends on the shape of material as in Table 9.1 in Appendix-A. As shown in this table, four general classes of shape have been found by Heywood (1971) with the statistical values for \( r_a \) and \( P_r \) for these classes. In addition, numerous measurements were made by Heywood (1971) (see Table 9.1) for the value of \( C \) that is used in Eq. (2.1.21). These relationships enable the surface area to be calculated from other properties such as \( n \), \( m \) and \( C \). Some examples of Heywood’s shape coefficient for different materials were shown in Appendix-A.

### 2.2 Packing Bed Properties

Packed beds or fixed beds have a number of applications in many operations such as adsorption, drying, filtration, dust collection, and other catalytic and non-catalytic reactors. One important reason to study a packed bed is to control the primary operating cost of the pressure drop through the packed bed of solids. Generally, voidage (\( \varepsilon \)) is the most frequently used and simple expression of packing characteristics that is determined by the geometrical arrangement of individual particles, for systems with a dense fluid like water, as in Eq. (2.2.1).

\[
\varepsilon = \frac{\rho_s - \rho_b}{\rho_s - \rho} \tag{2.2.1}
\]

While, for systems with negligible fluid density (e.g. taking measurements in air), this equation will be as Eq. (2.2.2).

\[
\varepsilon = 1 - \frac{\rho_b}{\rho_s} \tag{2.2.2}
\]

where, \( \rho_b \), \( \rho_s \), and \( \rho \) are apparent (i.e. bulk) density [kg m\(^{-3}\)], solid density [kg m\(^{-3}\)] and fluid density [kg m\(^{-3}\)] respectively. Particle volume fraction or packing fraction is \( C=1-\varepsilon \).
In a real powder process, size has a major effect on voidage, as open structures are more stable with smaller particles. The voidage increases with a decrease in the particle size. Although, uniform and regular mono-size spheres is the simplest case of a packed system, random packing of mono-sized spheres is more complicated and it can be described mathematically through a coordination number. Voidage of mono-sized spheres can be derived satisfactorily by mathematical considerations but, random packing of mono-sized spheres should be done through experiments (Masuda et al. 2007, Yang 2003). In the case of using binary mixtures of spherical and non-spherical particles, the diameter ratio is an important factor. It is found that when the diameter ratio decreases the changes in voidage increases. Furthermore, very little work has been done on the packing of non-spherical particles due to the complexity of such systems. Various shapes of particles have been examined experimentally and it is found that the more irregular the shape is the greater the voidage will be. Other researchers found that, the voidage results depend on the height of fall of the particles (Yang 2003).

2.3 Particle Porosity

Merkus (2009) and Holly et al. (1993) stated that, permeability cannot be defined in terms of porosity and an average pore size without considering the pore size distribution. The pore size distribution affects the flow pattern because the flow in a given pore does not only depend on the size of the pore being considered, but also on the size of the pores around it. At the same porosity the measured permeability and the simulated one are different, where the simulated is usually higher than the measured. It is found that, it is particularly difficult to establish a permeability model based only on the porosity (Ye et al. 2006). Scholes et al. (2006) maintained that, in most solid–liquid separation processes and fluid transport, flow characterisations through porous media are fundamental and anisotropy characterisation can affect permeability results. According to Merkus (2009) product quality is affected by the pores and their size distribution, which can either be good or bad. One example of a good effect is in the case of catalysts and adsorbents, where large pores facilitate easy access of reactants to active sites present in a large surface area provided within smaller pores.
2.4 Permeability

Permeability, according to Darcy’s law, can be defined as the rate at which a viscous fluid will flow through a porous medium at a given pressure gradient. Permeability measurement techniques can be classified depending on the method or the model used. The most commonly used methods to deduce permeability are permeation, filtration and sedimentation (Holdich 2002). It is common in data analysis to assume that there is no effect of sedimentation during cake formation. Hence, the cake resistance and permeability are calculated using equations based on Darcy’s law for liquid flow through a filter cake from graphical plots of time over filtrate volume plotted against filtrate volume (constant pressure filtration) or filtration pressure plotted against filtrate volume, or time (constant rate filtration). In general, these three regions namely: cake formation, transition and flow through the already formed cake region are present in most filtration tests (Couper et al. 2009, Li et al. 2005, Wakeman et al. 2005). As filtration proceeds, a porous cake of solid particles accumulates on a porous filter medium and its filtration rate can be found from the relationship below:

\[
\text{Rate of filtration} = \frac{\text{Driving Force}}{\text{Resistance}}
\]

By applying Darcy’s Law the flow rate can be found using Eq. (2.4.1) ignoring the negative sign needed to balance the vector quantities of filtrate velocity and pressure gradient.

\[
Q = \frac{dV}{dt} = \frac{A \Delta P}{\mu R}
\]  

(2.4.1)

where \(Q\) is the volumetric rate of flow \([\text{m}^3 \text{ s}^{-1}]\), and the pressure drop across the filter will be as shown in Eq. (2.4.2).

\[
\Delta P = \left(\frac{\mu R}{A}\right) \left(\frac{dV}{dt}\right)
\]  

(2.4.2)

where \(R\) is the total resistance \([\text{m}^{-1}]\) that is formed on the filter medium \((R_m)\) and the filter cake \((R_c)\). The total applied pressure \((\Delta P)\) on the system is equal to the
pressure drop over the cake ($\Delta P_c$) plus the pressure drop over the filter medium ($\Delta P_m$) as shown in Eq. (2.4.3).

$$\Delta P = \Delta P_c + \Delta P_m \tag{2.4.3}$$

From the cake filtration modelling, it can be assumed that Darcy's law can fit with both resistances (filter medium and filter cake) to fluid flow as shown in Eq. (2.4.4).

$$\Delta P = \frac{\mu R_c}{A} \frac{dV}{dt} + \frac{\mu R_m}{A} \frac{dV}{dt}$$

where $R_m = \frac{L_m}{k_m}$ the medium height ($L_m$) and its permeability ($k_m$) will be constant during the filtration. Similarly, the cake resistance ($R_c$) can be defined as $R_c = \frac{L_c}{k_c}$

Rearranging Eq. (2.4.4) will result in Eq. (2.4.5)

$$\frac{dt}{dV} = \frac{\mu}{\Delta P A} (R_c + R_m) \tag{2.4.5}$$

Although, the medium resistance is constant with time, the cake resistance will increase with the cake depth. Moreover, Eq. (2.4.5) has a lot of unknown parameters to solve. Cake resistance has a direct proportion to cake mass as shown in Eq. (2.4.6).

$$R_c = \alpha \frac{W}{A} \tag{2.4.6}$$

where, $\alpha$ is called the specific cake resistance to filtration [m kg$^{-1}$], $W$ is the total mass of the cake [kg], $W = cV$ and $A$ is the cake area [m$^2$]. At a constant feed concentration, and a known material, both the specific cake resistance ($\alpha$) and dry cake mass per filtrate volume ($c$) will be constants.

$$\frac{dt}{dV} = \frac{\mu \alpha c}{A^2 \Delta P} V + \frac{\mu R_m}{A \Delta P} \tag{2.4.7}$$

Eq. (2.4.7) is the general filtration equation that is valid for all types of incompressible cake filtrations. Addition of the filter cake and medium pressure drops provides the well-known linear equation for the analysis of constant pressure filtration (CPF) as illustrated in Eq. (2.4.8).
where $t$ is the filtration time [s], $V$ is the filtrate volume [$m^3$], $\mu$ is the liquid viscosity [Pa s], $A$ is the cross section filtration area [$m^2$], $c$ is the dry mass of solids per unit filtrate volume [$kg m^{-3}$], $R_m$ and $\alpha_{av}$ are ‘filtration constants’ that may be evaluated from the intercept and slope of the $t/V$ vs. $V$ plot, respectively. While, the equation for constant rate filtration (CRF) is shown in Eq. (2.4.9).

$$\Delta p = \frac{\mu \alpha_{av} c}{A^2} V + \frac{\mu R_m}{A \Delta p}$$  \hspace{1cm} (2.4.8)$$

In theory, the Eq. (2.4.9) will result in a linear pattern, provided that the average specific resistance and dry cake mass per unit volume of filtrate are constant in the case of incompressible filtration. The ‘filtration constants’ in Eq. (2.4.9) can then be found from the intercept and slope of a linear plot of $\Delta P$ against $V$, noting that $dV/dt$ is a constant, by definition. In the case of compressible cake filtration the Eq. (2.4.9) is still valid, at any increment in time, and the intercept can be used to determine the filter medium resistance, but the relation will deviate from linearity as specific resistance changes with the applied pressure over the filter cake. There are a number of methods which can be used to determine the dry cake mass per unit volume of filtrate ($c$). If the cake concentration by volume fraction can be independently determined, then it is possible to use the Eq. (2.4.10):

$$c = \frac{1}{(1-s)/(sp)-(1-C)/(CP_s)}$$  \hspace{1cm} (2.4.10)$$

where $s$ is the solid concentration of the slurry to be filtered as a mass fraction. The permeability of a filter cake is related to its specific resistance as shown in Eq. (2.4.11).

$$\alpha_{av} = \frac{1}{kC \rho_s}$$  \hspace{1cm} (2.4.11)$$

where $k$ is the hydraulic permeability [$m^2$], $C$ is the solid concentration by volume [$m^3 m^{-3}$] and $\rho_s$ is the density of solids [$kg m^{-3}$]. The specific resistance can be calculated
by using either Eq. (2.4.8) or (2.4.9) and then the permeability can be found using 
Eq. (2.4.11). A number of experimental techniques and analyses of the cake 
behaviour and structure have been published by many authors (Wakeman 2007, 
Tarleton 2001, Holdich et al. 1993, Rushton et al. 1984) and these indicate that within 
a filter cake it is normal to have a nearly uniform concentration throughout the cake, 
so that the incompressible filtration equations may be applied, using ‘average’ 
concentration and resistance (permeability) values. However, the solid concentration 
of the cake may differ with different filtration pressures. Hence, the occurrence of 
different values of cake concentration at different pressures does not invalidate the 
incompressible analysis of each individual filtration, so long as the appropriate value 
of concentration is used in that analysis.

Although, there are many numerical models already existing to predict permeability, 
Xu et al. (2008), the Kozeny-Carman (K-C) Eq. (2.4.12) and Happel-Brenner (H-B) 
Eq. (2.4.13) are the most widely used and accepted models.

\[
k = \frac{(1-C)^3 \psi^2 x_{sv}^2}{K C^2}. \tag{2.4.12}
\]
\[
k = \frac{(2-3C^{\frac{4}{3}} + 3C^{\frac{5}{3}} - 2C^2)}{(3+2C^{\frac{5}{3}})} \frac{x_{sv}^2}{12C}. \tag{2.4.13}
\]

where, \( k \) is porous media permeability [\( \text{m}^2 \)], \( C \) is solid concentration per volume (\( C=1-\varepsilon \)) \( \varepsilon \) is voidage, and \( x_{sv} \) is Sauter mean diameter [\( \text{m} \)] (\( x_{sv} = 6/ S_v \)), \( S_v \) is the specific 
surface area per unit volume [\( \text{m}^{-1} \)], \( \psi \) is the particle sphericity factor and \( K \) is called 
the Kozeny constant and approximates to values typically between 5 to 32 that 
generally depends on shape, size and porosity of particles (Tien et al. 2013, Mahdi 
2008, Holdich 2002). In the case of the K-C expression the shape of the particles 
may be included via a sphericity term (\( \psi \)) as shown in Eq. (2.4.12). The permeability 
could be converted to specific resistance using Eq. (2.4.11). From this analysis 
average permeability, or cake resistance, values can be deduced and compared with 
the permeability models, but the data and research to-date appears to suggest that it 
is unlikely that a single analytical equation such as Eq. (2.4.12) or (2.4.13) will be 
valid to correlate permeability (or cake resistance) with particle properties, Tien et al. 
(2013) and Rushton et al. (2000), for all systems encountered within Chemical 
Engineering principles and applications.
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The K-C model is based on the entire particle size distribution, particle shape (sphericity factor) and void ratio or solid concentration, it is also one of the most accepted and used definitions of permeability as a function of the characteristics of the medium. It is applied in various fields such as ground water flow, water/oil reservoirs, chemical engineering, medicine, biochemical and electro-chemical engineering. Also, it is widely used as a starting point for many other permeability models (Xu et al. 2008). Darcy’s Equation or Law is further developed by Kozeny who assumed that a granular bed is the same as a group of similar, parallel ducts such that the total internal surface and volume are equivalent to the particle surface and pore-volume in the packed bed, respectively.

It has been calculated that for two sizes of mixture of spherical particles when the size ratio goes over 4:1 and the percentage of smaller spheres in the mixture is less than 40% the Kozeny model does not fit (Carman 1956). Carman (1939) reported that, permeability depends only upon the size, shape and packing mode of the particles forming a bed of stiff particles. The K-C model has also certain limitations in which it is not suitable for very fine materials such as clay, because it assumes there are no electrochemical interactions between materials and fluid, it is appropriate with just laminar flow and low fluid velocity, it cannot work with extreme particle size distribution that has a long or flat tail in the fine fraction and it does not account for isotropy. Practically most work done using this model uses vertical permeability, which is less than the horizontal one (David et al. 2003). Using the K-C equation to predict the permeation resistance of either gas or liquid passing through particle layers, Endo et al. (2002) investigated the considerable error that is encountered when this equation is used with particle beds having a wide particle size distribution and/or non-spherical particle shape. According to Peker et al. (2008) and Svarovsky (2000) one of the most important limitations of the K-C model to predict permeability is that, it is only valid when the porosity is less than 0.6.

As a result of these limitations of the K-C model, several researchers have attempted to derive a model with a more realistic and general outcome. One significant attempt was the free surface cell model by Happel-Brenner (Eq. (2.4.13)) in which each particle is assumed to be a sphere and the porosity of each cell is the same as that of the bed. Happel’s cell model takes into consideration the drag forces acting on the
individual spheres. This Happel solution provides a better description of the microscopic flow field through a structure of interacting, but single and mono-sized spheres, to the Navier–Stokes equation (Happel et al. 1965).

2.5 Computational Fluid Dynamics (Permeability Simulation)

A detailed knowledge of flow phenomena within a packed bed is useful in many industries e.g. oil recovery, petrochemical, paper making, fine chemical and pharmaceutical. However, in the case of studying heat and mass transfer characteristics, an understanding of interstitial flow in the void space is required. One of the most important numerical methods to investigate the complex behaviour of a fluid through a packed bed is Computational Fluid Dynamics (CFD). There are many advantages of using a CFD model such as the relatively low setup cost and less work-hours for accepted accuracy. The CFD approach appears to be more general than others (Baker et al. 2010, Tobis 2008, Gunjal et al. 2005). In addition to that, Jafari et al. (2008) and Calis et al. (2001) also reported that CFD simulation results can be correlated well with experimental data. In general, the flow can simply be described by Darcy’s law Eq. (2.5.1), which is valid for sufficiently low Reynolds number, incompressible and Newtonian fluid and a fixed porous medium.

$$\frac{\Delta P}{L} = -\frac{\mu Q}{kA} = -\frac{\mu}{k} U_0$$ (2.5.1)

where $k$ is the bed permeability $[m^2]$, $\mu$ is the viscosity of the fluid $[Pa\ s]$, $Q$ is the flow rate $[m^3\ s^{-1}]$ through an area $A\ [m^2]$, $\Delta P$ is the pressure drop $[Pa]$ over a length $L\ [m]$ in the stream direction and $U_0$ is the superficial velocity $[m\ s^{-1}]$. The modified Reynolds number ($Re'$) Eq. (2.5.2) is:

$$Re' = \frac{U_0 \rho}{S_v (1-\varepsilon) \mu}$$ (2.5.2)

where $\rho$ is the fluid density $[kg\ m^{-3}]$, $S_v$ is the specific surface area $[m^{-1}]$, $\varepsilon$ is the bed voidage. Baker et al. (2011) and Baker et al. (2010) concluded that based on the number of particles packed into a known volume the bed can be loosely or densely packed and this is sometimes referred to as packing efficiency. In addition, there are two different types of packing, which are random (unstructured) and structured where
the coordinates of each particle have a full mathematical description and constant respective porosities and packing densities. However, in the case of random irregular size/shape particles none of these are constant. They also investigated two different packings of mono-sized spheres that are simple cubic and face centred cubic packing. It was found that face centre cubic packing was highly dense compared to the others but they did not provide any mathematical proof. Happel et al. (1965) stated that, to control boundary value problems containing a number of particles, two CFD techniques can be employed that are either reflections or the unit cell (UC) approach. The reflection approach has been used by a number of researchers, Baker et al. (2010), Jafari et al. (2008) and Calis et al. (2001), but it is still not the most favourable approach for others. Generally, using the full packed (reflection) bed model simulation with CFD a pre-processor stage of defining a suitable geometry in which the fluid flow problem is to be analysed is required. This stage can be achieved by replicating a known geometry through Computer Aided Design (CAD) (Baker et al. 2011, Baker et al. 2010).

On the other hand, in the UC approach, rather than studying the whole bed, only one or two porous media are studied and the flow is assumed to be the same in all the other pores contained within the bed. Happel et al. (1965) investigated the UC approach and they did establish their own cell model for predicting permeability Eq. (2.5.3) based on bed voidage \(( \varepsilon = 1 - C )\) and particles specific surface area \(( S_v = 6/x_{sv})\).

\[
k = \frac{(2 - 3C^{1/3} + 3C^{5/3} - 2C^2)}{(3 + 2C^{5/3})} \frac{x_{sv}^2}{12C}
\]  

(2.5.3)

where \( C \) is the cake concentration by volume fraction and \( x_{sv} \) is the Sauter mean diameter [m] of the particle distribution. This approach is applied by assuming a regular structure to the bed which can be reproduced using a simple UC. The UC technique involves the concept that an assemblage can be divided into a number of identical cells where each cell contains one particle. Also, a number of studies have employed different particle and cell shapes. The assumption of a spherical shape for the particle and for a fictitious envelope of fluid surrounding is of great significant as a sphere can be described in terms of a single parameter (Hellström et al. 2006, Sorensen et al. 1995, Happel et al. 1965). The two main reasons for using this
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approach are the relatively low mesh sizes and less computational processing time, which is not the case when using many particles (Calis et al. 2001).

Furthermore, the UC method has been adapted by many researches. Tobis (2008) compared both experimental and numerical results of air turbulent flow through six similar structured beds of spheres in a simple cubic unit cell. The results show that, both values are in a good agreement. Also, by using a single UC approach of an array of spheres in different periodically repeating arrangements of particles, Gunjal et al. (2005) investigated the interstitial fluid flow in the void space. Also, it was used by Hellström et al. (2006) to perform a micro-mechanically based study of moderate modified Reynolds number (Re’) flow between parallel cylinders in order to increase the understanding of such flow by comparing the results with an empirically derived Ergun equation. They reported that the Ergun equation fits well with simulated data from Re’=20 and the steady calculations can be used up to Re’=880. Durst et al. (1987) simulated laminar flow through UC and found that the simulated pressure results agreed with the experimental data.

The UC approach is found to be useful for understanding the flow characteristics in a packed bed (Gunjal et al. 2005). This approach applies strictly only to periodic arrays and in some stochastic sense to random particle arrays, where wall container effect is neglected (Happel et al. 1965). The focus of the work presented in this thesis is to study different kinds of packed beds permeability. Both results from the CFD model and analytical solution using H-B model will be compared. Also, the phenomena of water fluid flow through packed beds and how the bed geometry and packing structure affects the flow will be investigated. The main objective of using CFD in this work is to create a simulation model in order to generate permeability values under different conditions, e.g. particle shape. These data will be used later with the numerical models (ANN and MVR) for predicting permeability instead of using an experimental lab that requires more time and cost.

2.6 Computing Methods (Permeability Prediction)

In practice, permeability is a function of various parameters including particle size, particle shape, voidage and packing arrangement, particle size distribution, and in conditions where the concentration of slurry is being filtered (Hakkinen et al. 2008,
Rushton et al. 2000). Hence, theoretical relations for permeability are often used as a guide to estimate permeability if no operating data is available. Measured permeabilities may be one or even two orders of magnitude lower than that given by the analytical models (Wakeman 2007, Yim et al. 2001).

There is a need to develop a new model that will predict permeability more efficiently than the present models. There is a big number of application programmes that could fit in this case. Two different prediction methods are used in the current work: Multivariate Regression (MVR) and Artificial Neural Network (ANN). The ANN approach was chosen here because of its applications in solving complex problems particularly when dealing with nonlinear systems. The parallel computation and learning abilities of neural networks make them an alluring tool in many chemical engineering problems. The only requirement to guarantee optimal performance is a series of carefully selected examples of desired input/output relations (Haykin 2009).

In MVR, there are a number of different models that can be used, but principal component regression (PCR), partial least squares regression (PLSR) and simple nonlinear regression (NLR) are the common models and hence were chosen. All of these methods (PCR, PLSR and NLR) are predictive models building technique (Saleemi 2011). In many computing models pre and post-processing steps are required, which leads to an improvement in the model training efficiency. There are several routines that can be used; the most common are provided automatically from the model. In general, both the input and the output vectors are normalized before applying the regression algorithm (Saeedi et al. 2007).

### 2.6.1 Multivariate Regression

The MVR model is one of the most widely used of all statistical methods. MVR accommodates more than one response variables and that can be useful when variability in the independent variable occurs. Also, MVR models such as PCR and PLSR, are based on the inverse method (Gosasang et al. 2011, Fox et al. 2011, Bakeev 2010). The PLSR and PCR approaches are both linear and similar with the only main difference, which is the way that the data is compressed. In PCR the regression is applied to those variables that account for variance in the input data, but in PLSR is variance present in both (input and output) and this is considered during model building. As a result, the compressed variables obtained will be
different and are termed as latent variables (Bakeev 2010, Gemperline 2010). The MVR general linear model is shown in Eq. (2.6.1).

\[ k = bX + e \]  

(2.6.1)

where \( k \) is the predicted permeability, \( X \) is an \( n \times m \) matrix of multiple variables such that each row corresponds to a complete spectra recorded at \( m \) wavelengths. A column of ones can be introduced to account for the intercept term; in this case \( X \) will have \( n \times (m+1) \) dimensions. The vector \( b \) (\( b = [b_0, b_1, \ldots, b_n] \)) has all the regression coefficients, each corresponding to a specific variable present in the \( X \) matrix, \( e \) is independent (errors). In case of using PCR and PLSR the prediction equation for both models will be the same as shown in Eq. (2.6.2).

\[ k = tb + e \]  

(2.6.2)

where \( t \) is the principal score vector and it can be calculated using Eq. (2.6.3)

\[ t = \tilde{X}P \]  

(2.6.3)

The prediction steps for PCR and PLSR are similar. However, PLSR is more complex than PCR as it uses information simultaneously from dependent and independent variables. Also, it has different steps to calculate the regression coefficients as in Eq. (2.6.4).

\[ b = W \left( P^T W \right)^{-1} g^T \]  

(2.6.4)

where \( T \) is the latent matrix and \( P \) is the principal components matrix that is found from Eq. (2.6.5).

\[ P^T = W^T \tilde{X}^T X \]  

(2.6.5)

where \( \tilde{X} \) is the responses and \( W \) is the eigenvector corresponding.

In a number of studies comparing MVR and ANN models the values of root mean square error (RMSE) have been used to determine which technique fits best. In general, the ANN models showed better fit as it is capable of catching sophisticated
non-linear integrating effects (Gosasang et al. 2011, Moradzadehfard et al. 2011, Pao 2008). In one comprehensive study, Saleemi (2011), the PCR model failed to predict the output accurately and a significant difference was observed between the measured and predicted values. In general, the MVR model faces difficulties when multicollinearity exists in the data. Multicollinearity occurs when some of the variables can be expressed as linear functions based on other variables in the system. This limits the MVR capability and leads to an unstable model with poor predicted response (Saleemi 2011). Johnson et al. (2007) reported that the published applications of multivariate methods have increased tremendously in recent years to include data reduction or simplification, sorting and grouping, investigation of the dependence among variables, prediction and hypotheses testing.

2.6.2 Artificial Neural Network

The ability of ANN to learn from existing data makes it a potentially very useful computer tool. The ANN eventually learns the correct behaviour, and can reproduce correct outputs on its own by training with suitable input data (Haykin 2009). Many authors, Zargari et al. (2013), Pazuki et al. (2012), Gurney (1997) and Krose et al. (1996), defined ANN as an interconnected assembly of simple processing elements (units or nodes) whose functionality is based on the animal neuron and learning from a set of training patterns. Unlike MVR despite performing data regression, ANNs do not need to identify structural correlations between input and output data.

A number of researchers Zargari et al. (2013), Brownlee (2011), Haykin (2009), da Silva et al. (2008), Iwata et al. (2007) and Boroumand et al. (2005) describe ANN as consisting of nodes operating in parallel as shown in Figure 2.3. These nodes are in three general layers, which are input, hidden and output. The data enters the first layer then moves to the hidden layer where summation functions, which could be one of: sum, max, min, average, or, and, etc. After that it moves to the transfer function layer, which includes hyperbolic transfer, linear, sigmoid, sine, etc. Finally the network presents the data in the form of output information. The network function is determined from the connections between the nodes. An ANN can be trained to perform a particular function by adjusting the values of the connections (weights) between nodes and the particular input leads to a specific target. The network
compares the output to the target, until the network output matches the target (Zabihi et al. 2011, Wang 2006, Shetty et al. 2003).

![Nonlinear Model of a Neuron](source-haykin-2009)

Figure 2.3: Nonlinear Model of a Neuron
Source: Haykin (2009)

Each input in ANNs is multiplied by a weight before being sent to the next layer. A simple arithmetic addition is used to sum the weighted signals to supply node activation and the results are compared with the Threshold Logic Unit (TLU). The unit produces a high-valued output of ‘one’ if the activation exceeds, otherwise it produces an output of zero (Haykin 2009, Iwata et al. 2007, Anderson et al. 1992). An ANN model is still designed in a time-consuming iterative trial and error method, which depends mostly on the problem itself and the programmer’s experiences. However, the technique of virtual approximation of correlations has improved and the approach is applicable for prediction of various properties (Boozarjomehry et al. 2001).

**Topology (Architecture) of ANNs**

There are two different classification levels: internal and the external structures. The internal structure describes the connections between individual neurons both within and between layers. However, the external one describes the overall connections between input, output and hidden layers. In general, all ANNs are based on the concept of neurons, connections, and transfer functions (Haykin 2009, Boroumand et al. 2005, Hong et al. 2003, Anderson et al. 1992). Also, in ANNs there are two different kinds of network which are the single-layer and the multilayer network (Christos et al. 2010). The multilayer networks can either be Feed-Forward function (FF) or Feed-Forward Back-Propagation (FFP), (Brownlee 2011, Christos et al. 2010, Haykin 2009, Wang 2006). In FFP function, data is recycled from the transfer
functions into the hidden layer in order to adjust the weight of the inputs and recalculating the output according to the new values, (Iwata et al. 2007, Anderson et al. 1992). ANNs with the FFP algorithm are the most popular and heavily used (Nowroozi et al. 2009, Saeedi et al. 2007, Arpat et al. 1998).

**Transfer Functions**

Although, many transfer functions can be used with ANN, Khataee et al. (2010) Haykin (2009) and Boroumand et al. (2005), the most commonly used are log-sigmoid, tan-sigmoid and linear (see Appendix-C). For a linear function, neurons are used in the final layer of multilayer networks as function approximations. The input can have any value between ± infinity as correlated by the sigmoid function, which transposes the output into the range of 0 to 1, (Khataee et al. 2010, Haykin 2009, Boroumand et al. 2005).

**Training, Validation and Testing the ANNs**

The basic stage of an ANN approach is a training stage. It is used to adjust the network according to its error, computing the gradient and updating the network weights and biases. There are different algorithms for training, and it is difficult to identify the fastest and most accurate one (Pazuki et al. 2012, Haykin 2009). Supervised and unsupervised training methods are used (Sinha et al. 2007, Boroumand et al. 2005, Jeff 2005). However, a supervised training algorithm has the most applications (Zabihi et al. 2011, Arpat et al. 1998). In order to improve the model performance, a high number of data points need to be used for the training set (Jeff 2005). It is normal to test and validate the performance of the ANN after training is finished (Haykin 2009).

**Advantages and Disadvantages of ANNs:**

ANNs Advantages:

The main advantages of ANNs are: it only requires basic level programming as packages are available, it solves complex problems (non-analytical and/or nonlinear
and/or non-stationary and/or stochastic), a self-organizing and learning mechanism and applies to a wide range of problems without re-programming. It is becoming widely accepted to simplify programming and algorithm design for a given wide range of outputs (Christos et al. 2010, da Silva et al. 2008, Graupe 1999). ANNs are particularly useful for solving problems that cannot be expressed as a series of steps, such as recognizing patterns, classifying into groups, series prediction and data mining, (Christos et al. 2013, Jeff 2008). The utilization of the ANN has been reported in the prediction of permeability for the purposes of petroleum reservoir engineering, Tahmasebi et al. (2012), but this was not based on particle characterisation such as size distribution and shape.

**ANNs Disadvantages:**

According to Agachi et al. (2006) ANNs have limited ability to identify possible causal relationships. This is because no well-established criteria exist for interpreting the weights and biases. When dealing with a large number of variables ANNs are likely to over fit and the computation becomes difficult. Hence, more time is required during the computation and optimization of the complex processes (Zargari et al. 2013, Jeff 2008).

Permeability is one of the most difficult physical properties to predict. It can be time and cost consuming to obtain from laboratory data and is prone to subjective interpretation (Zargari et al. 2013). Instead of using traditional regression techniques, ANNs provide accuracy, consistency and improved overall quality of permeability prediction for reservoir engineering (Wiener et al. 1995). Wakeman et al. (2003b) established a relationship for the specific cake resistance and the combined (vibration) cake resistance using an ANN model. This model consists of three inputs (vibration acceleration, cumulative filtrate and concentration), using one hidden layer (8 neurons) and one output (vibration specific cake). They found that ANNs showed promising results, but this method also has its own disadvantages such as failing to succeed when limited analysis data are available (Arpat et al. 1998). In general, using a large number of data points increases the processing time and decreases the number of missed data (Tahmasebi et al. 2012). More recently, Pazuki et al. (2012) studied the efficiency and accuracy of the ANN model for prediction of oil reservoir
permeability as a key parameter in reservoir engineering. Various Multilayer Perceptron (MLP) models, with different learning algorithms, layers and node numbers, were investigated. Their results show that, ANN with Improved Back Propagation (IBP) learning method and five nodes in the middle layer gave the highest accuracy for their applications.

In order to make a rational decision about methods of computational intelligence Zargari et al. (2013) compared predicted permeabilities, again for the purposes of reservoir engineering, from ANN and Adaptive Neuro-Fuzzy Inference System (ANFIS). Results show that, the ANN model was more accurate than the ANFIS. Tahmasebi et al. (2012) investigated the performance and accuracy of two different permeability prediction methods: Modular Neural Network (MNN) and traditional Multilayer Perceptron (MLP). The obtained permeability results showed that incorporating MLP showed good prediction. In the case of soil compaction and groundwater engineering Sinha et al. (2007) concluded that ANN prediction models could be used for compression and permeability determination with sufficient accuracy for their purposes, but again this was not work based on fundamental particle characterisation. Therefore, it is apparent that ANNs have been accepted widely as tools for research in petroleum reservoir engineering and Geotechnical applications.

In the above work, researchers used different input parameters e.g. electrical conductivity and resistivity, photovoltaic effect, solid density, bulk density, sonic transient time, spectral Gamma ray, deep induction log, correlated log porosity, depth and water saturation. These parameters describe the local properties of the reservoir rather than the solid itself. Also, none of these parameters have considered either the particle shape or size distribution. Their analysis was appropriate for the modelling of a large consolidated reservoir, but not for the constituents forming the reservoir.

**2.6.3 Model Performance**

The efficiency of network design mostly depends on learning algorithm, topology and data distribution, which change from one dataset to another (Tahmasebi et al. 2012). The criteria used to evaluate the performance of a model usually are the Coefficient of Correlation ($R^2$), Root Mean Square Error (RMSE) and Average Absolute Error
(AAE) as shown in Eq. (2.6.6), (2.6.7) and (2.6.8) respectively (Pazuki et al. 2012, Sinha et al. 2007).

\[ R^2 = 1 - \frac{\sum(Y_{\text{measured}} - Y_{\text{pred}})^2}{\sum(Y_{\text{measured}} - \frac{\sum Y_{\text{measured}}}{N})^2} \]  

(2.6.6)

\[ RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N}(Y_{\text{measured}} - Y_{\text{pred}})^2} \]  

(2.6.7)

\[ AAE = \sum \frac{|Y_{\text{measured}} - Y_{\text{pred}}|}{Y_{\text{measured}}} \]  

(2.6.8)

where, \( Y_{\text{pred}} \) is the network prediction value, \( Y_{\text{measured}} \) is the experimental response value, \( N \) is the total number of readings in the data points. RMSE is the basic tool to check the model accuracy (Khataee et al. 2010, Haykin 2009, Shetty et al. 2003). These three measures of performance were applied for both numerical methods (MVR and ANN) while AAE was used to evaluate the analytical models (K-C and H-B).

The purpose of the research reported here is to develop an alternative approach for the estimation of porous media permeability (and therefore specific resistance or any other derivative of permeability) for various materials under different conditions from particle characterisation data and process parameters and relevant to non-consolidated systems such as during filtration and solid-liquid separation. This alternative approach can be employed for design and modelling purposes and is based on statistical computational techniques (MVR) and Artificial Neural Network (ANN) modelling.
2.7 Concluding Remarks

Particle characterisation is an important aspect of manufacturing processes. The particle characterisation is required in order to understand the behaviour of the particle. There are many methods which can be used to characterise the particle but there are still some limitations existed. The Heywood shape coefficient method was discussed in detail in this chapter and applied to investigate the particles because of two reasons. One is the accuracy of this method to calculate shape coefficient and secondly to enable this method to be used in mainstream techniques.

The packed bed has a wide range of applications, therefore understanding the pressure drop phenomena is an important aspect in order to control the primary operating cost. It is found difficult to establish a model of permeability prediction that is based only on porosity, or voidage. The size and the orientation of particles during mechanical compaction is affecting the permeability results and makes it more difficult to calculate theoretically. Although there are many different analytical models to determine permeability, the K-C and H-B models are the most widely used with several limitations. The concentration of particles affects the particle bed permeability, which decreases when solid concentration is increased. Furthermore, measurement of permeability is interrupted by some parameters that affect the permeability value, which includes the tortuosity ratio, anisotropy, size distribution and shape of particles. CFD is an important numerical method to investigate the complex behaviour of a fluid through the packed bed, having comparatively low setup cost and requires less work-hour with accepted level of accuracy. It is found that, the unit cell (UC) approach is a widely used model based on CFD to predict the porous media information. The low meshes and less computational processing time are the two significant reasons of using this approach. The purpose of using the CFD in this work is to simulate permeability in packed bed in order to generate permeability values under different conditions. The simulated data will be used later with the numerical models (ANN and MVR) for predicting permeability instead of using experimental lab, which is both time consuming and not high cost.

A number of statistical tools can be used to determine permeability value. However, the MVR within Excel and ANNs based on MATLAB have been used for a long time and therefore were chosen in this work. Both tools have some disadvantages as well
as some advantages, which are more beneficial than the disadvantages. In order to design a model there are standard steps that must be followed as will be discussed later in Chapter Four. Many different ANNs can be designed and used depending on the degree of the problem. As the process becomes more complex the number of neurons, layer and functions increases. Comparative analysis of the model is important to check its accuracy. Model performance is one of the known widely used methods to evaluate the degree of accuracy of any model and its level of acceptability.
CHAPTER THREE

MATERIALS CHARACTERISATION

This chapter explains the material characterisation in order to give a clear understanding of each material investigated. Particle size, size distribution and shape analyses were performed using different methods such as Scanning Electron Microscope (SEM), Laser Diffraction (LD) and Image Analysis (IA). For all solids, compressibility and particle surface charge were investigated under various applied conditions.

Three different materials were investigated experimentally to study various systems having a variety of characteristics such as shape and size distribution. Suspensions of talc (Mg₃Si₄O₁₀(OH)₂, White-Talc MINELCO) at different solid concentrations: 0.01, 0.02, 0.03, 0.04 and 0.05 (v/v) were prepared and represented a compressible system. For an incompressible system suspensions of aqueous calcium carbonate (CaCO₃, Fordacal-30 MINELCO) at 0.09, 0.11, 0.13, 0.15, 0.17, 0.19 and 0.21 (v/v) of solid concentration were used. Each suspension was prepared by dispersing the dry powder in reverse osmosis water (RO-water). Also, titanium dioxide P25 (TiO₂-AEROXIDE®, with primary particle diameter of 21 nm) was used as a colloid system in order to study the performance of clusters of particles using an initial concentration range from 0.003 to 0.03 (v/v). P25 was mixed with RO-water containing the electrolytes sodium nitrate, NaNO₃, (Fisher Scientific UK Ltd) and magnesium sulphate, MgSO₄, (Sigma–Aldrich Co.) as coagulants. The amount of both coagulants (1 mM each) was selected based on equalling the ionic strength of the local tap water. Initial tests of P25 in tap water showed good separation of the two phases, and clarity of the supernatant.

To avoid the ionic variation found in tap water, RO-water was used instead with all solids, with added electrolytes where appropriate. The natural pH was constant for all tests of different solids. Sauter mean diameter and size distributions of particles were calculated using Malvern 2000 and Horiba LA-920 equipment. The Multivolume Pycnometer 1305 was used to measure density of the materials. Also, scanning electron microscope (SEM) images for dry powders were taken as shown in Figure 3.1. All results were recorded and tabulated as shown in Table 3.1.
Figure 3.1: Scanning electron microscope (SEM) images of talc (a), calcium carbonate (b) and titanium dioxide P25 (c).
Table 3.1: Relevant properties and characterisation information of the used materials

<table>
<thead>
<tr>
<th></th>
<th>Talc (1)</th>
<th>Calcium Carbonate (2)</th>
<th>Titanium Dioxide (P25) (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha_0$ (m kg$^{-1}$)</td>
<td>1.1x10$^3$</td>
<td>6.6x10$^3$</td>
<td>6x10$^-1$</td>
</tr>
<tr>
<td>$n$</td>
<td>0.47</td>
<td>0.19</td>
<td>0.35</td>
</tr>
<tr>
<td>$C_0$ (v/v)</td>
<td>0.17</td>
<td>0.25</td>
<td>0.022</td>
</tr>
<tr>
<td>$m$</td>
<td>0.21</td>
<td>0.08</td>
<td>0.14</td>
</tr>
<tr>
<td>Natural pH</td>
<td>9.4</td>
<td>8.0</td>
<td>5.3$^5$</td>
</tr>
<tr>
<td>Zeta potential (mV)</td>
<td>-13.9</td>
<td>+11.1</td>
<td>+139.5</td>
</tr>
<tr>
<td>IEP point</td>
<td>pH=2.0</td>
<td>pH=10.3</td>
<td>pH=6.3</td>
</tr>
<tr>
<td>S.M.D (4) (µm)</td>
<td>7.0</td>
<td>7.5</td>
<td>2.5</td>
</tr>
<tr>
<td>Density (g.cm$^{-3}$)</td>
<td>2.978</td>
<td>2.790</td>
<td>4.034</td>
</tr>
</tbody>
</table>

(1) Initial concentration 0.01 to 0.05 (v/v) applied pressure range 14 to 67 kPa
(2) Initial concentration 0.09 to 0.21 (v/v) applied pressure range 8 to 80 kPa
(3) Initial concentration 0.007 to 0.03 (v/v) applied pressure range 16 to 67 kPa
(4) S.M.D is the Sauter mean diameter (in microns) was measured using a Horiba LA-920-wet LD.
(5) This pH is after adding the coagulants, however, the original natural is pH=2.5

3.1 Zeta potential measurements

The measurements of the pH and the Zeta potential were performed on the Acoustosizer IIS™ by using a potentiometric series of tests at room temperature (20 ±2°C) using 150 ml of RO-water for each solid. As shown in Figure 3.2, although, the Zeta potential of calcium carbonate (calcite) is positive and relatively low (-5 to +15 mV), it is negative for talc (up to -20 mV). In general, calcite is a system where surface effects are relatively small (Wakeman et al. 1991). According to Gribble et al. (2010) the structure of talc consists of (magnesium hydroxide, MgOH$_{20}$) layer sandwiched between two sheets of silica (SiO$_2$). These layers of talc are approx. 19 Å thick and they are held together by weak van der Waal's forces. The edges of talc are predominantly hydrophilic hydroxide groups, whereas the faces have hydrophobic groups. Hence, the measured Zeta potential is the net charge over the differing surfaces and the mineral can form structures commonly known as a ‘house of cards’ similar to clays.

However, the Zeta potential of titanium dioxide (P25), which is colloidal material with nanosize particles, ranging from -450 to +430 mV (see Figure 3.2). Cao et al. (2011) and Tarleton et al. (2003) reported that, finer particles (less than 10 µm) are affected by repulsive inter-particle force. This is affected by the ionic strength of the solution and increases with both solid volume fraction and Zeta potential in the mixture. Inter-
particle force is very low around the isoelectric point (IEP) and particle aggregation occurs easily and settles more quickly. As a result, the process engineer can expect faster settling rates, rapid filtration and higher moisture content cakes. Both the compaction rate and the final compact porosity are affected slightly by the suspension pH (Wakeman et al. 1993). Comparing the measured results with the supplier ones for all solids, both are similar. The results for the three solids are illustrated in Table 3.1.

![Graph of Zeta potential vs pH for Calcite and Talc](image)

Figure 3.2: The variation of the Zeta potential of solids used with pH

### 3.2 Laser Diffraction size analysis

A series of tests at different analytical flow cell pump speeds within the Laser Diffraction (LD) equipment are reported with respect to the analysis time. The circulation system employs a pump with variable speeds corresponding to different shear values. An analysis at different shear rates helped to illustrate both the kinetics of the clustering and the identification of the equilibrium cluster size. Figure 3.3 shows the variation of $x_{10}$, $x_{50}$ and $x_{90}$ of particle diameters over time for talc and calcium carbonate. Three different shear rates induced by increasing the pump speed (P.Sp.) were used during the LD analysis using the Horiba LA-920 instrument. The samples were subjected to various shears and sonication energy in order to monitor the formation of aggregates. Ideally, in-situ cluster analysis is required during hindered sedimentation. However, the relatively high concentration of dispersed phase and the consequent light scattering problems made this difficult hence clustering at low concentration was tested instead. This work demonstrates no evidence of significant aggregation or clustering on talc and calcium carbonate under the prevailing conditions. On the other hand, for titanium dioxide the evidence of how
easy the clusters can form, is provided in Figure 3.4. As shown in this figure each curve corresponds to the kinetics of clustering at a specific value of shear. Only at the lowest shear value did clustering occur to a significantly greater value than that initially formed: the measured Sauter mean diameter varied from an initial value of approximately 2 μm up to about 10.8 μm depending on the shear conditions.

At higher shear, lower equilibrium diameters were obtained, as large clusters were prevented from forming. At the highest shear, the initial diameter was constant over the entire time investigated at a value of approximately 2.5 μm. The suspension ionic strength was controlled by using filtrate water within the Horiba instrument to ensure similar conditions (ionic strength composition) between the characterisation and filtration experiments. This particle characterisation demonstrates that talc and calcium carbonate differ in their clustering behaviour.
calcium carbonate (calcite), have very similar median particle size and distributions as determined by the usual reported values of $x_{10}$, $x_{50}$ and $x_{90}$, based on a mass (or volume) particle size distribution. While, P25 is much smaller in size and it easily clusters.

![Figure 3.4: Variation of the Sauter mean diameter over time for five different values of shear fields created by increasing pump speed from 1 (lowest value) to 5 (highest) during the LD analysis on titanium dioxide.](image)

### 3.3 Compressibility tests

In order to determine the values of the coefficients in the constitutive equations for scale-up constants, a series of runs over the pressure range of 14 to 67 kPa for talc, 8 to 80 kPa for calcium carbonate and 16 to 67 kPa for P25 were performed. Tiller et al. (1983) concluded that, material compressibility ($n$) value of 0.2 is only a slightly compressed cake. Based on that statement, talc is a compressible system and calcium carbonate is relatively incompressible system used in this work, but with similar ‘average’ particle size. While, P25 material has very different physical properties compared to the others, dependent on shear. A summary of the empirical constants obtained through consistent analysis procedures indicates the reasonably compressible nature of the three materials is shown in Table 3.1. Both the average cake resistance and cake concentration can be found by applying the values of $\alpha$, $n$, $C_0$ and $m$ from Table 3.1 in Eqs. (3.3.1) and (3.3.2) respectively.

$$\alpha_{av} = \alpha_0 (1 - n) \Delta P^n_p$$  \hspace{1cm} (3.3.1)
\[ C_{av} = C_v (1 - m) \Delta P_c^m \]  

\[(3.3.2)\]

### 3.4 Shape of Particle

Applying the flowchart of Heywood’s steps (Figure 4.5) on different kinds of materials such as talc, calcium carbonate, P25, sand and glass beads etc., Table 3.2 was constructed. Moreover, different types of shapes were used in order to give a clear description of shape coefficient in each shape class.

<table>
<thead>
<tr>
<th>No</th>
<th>Shape Type</th>
<th>Shape Picture</th>
<th>Shape Coefficient</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cube: Square</td>
<td></td>
<td>+0.5</td>
</tr>
<tr>
<td></td>
<td>Rectangle</td>
<td></td>
<td>0.25 - 0.4</td>
</tr>
<tr>
<td>2</td>
<td>Sphere</td>
<td></td>
<td>+0.5</td>
</tr>
<tr>
<td></td>
<td>Half Sphere</td>
<td></td>
<td>0.4 - 0.5</td>
</tr>
<tr>
<td>3</td>
<td>Prism</td>
<td></td>
<td>0.2 - 0.3</td>
</tr>
<tr>
<td></td>
<td>Short</td>
<td></td>
<td>0.3 - 0.4</td>
</tr>
<tr>
<td>4</td>
<td>Cylinder</td>
<td></td>
<td>0.2 - 0.4</td>
</tr>
<tr>
<td>5</td>
<td>Rod Cylinder</td>
<td></td>
<td>0.1 - 0.4</td>
</tr>
<tr>
<td></td>
<td>Cube</td>
<td></td>
<td>0.1 - 0.2</td>
</tr>
<tr>
<td>6</td>
<td>Needle</td>
<td></td>
<td>0.1 - 0.2</td>
</tr>
<tr>
<td>7</td>
<td>Flake</td>
<td></td>
<td>0.01 - 0.1</td>
</tr>
<tr>
<td>8</td>
<td>Flake (Plate)</td>
<td></td>
<td>0.001 - 0.01</td>
</tr>
</tbody>
</table>

Table 3.2: Shape coefficient value of standard particle shapes

It is found that the most important region of shape coefficient is the one that is less than 0.5 where most of the particle coefficient shape values are. The decision was made to divide shape coefficient into seven categories: 0.001 to 0.01, 0.01 to 0.1, 0.1 to 0.2, 0.2 to 0.3, 0.3 to 0.4, 0.4 to 0.5 and +0.5, which is higher than 0.5. From Table 3.2 it can be seen that, the shape coefficient value is high for a sphere or
spherical object and it is low with flake or plate like shapes. It is assumed that permeability values have a proportional relation with the shape coefficient of the particle and this will be tested later in this thesis. Table 3.2 can be used to find the shape coefficient for different particle shapes.

3.5 Measure of Spread \((x_{50}/x_{10})\)

Malvern Mastersizer 2000 and Horiba LA-920 equipment were used to determine Sauter mean diameter for all solid materials, which have been used in calculations and the full size distribution. In the Horiba LA-920 sonication and mixing were used to avoid, or track, cluster size. Both \(x_{50}\) and \(x_{10}\) diameters were determined from the analyses. It was found that the range of this parameter is between 1.15 and 29.41. This high value of spread (29.41) shows that there is a high variation in the sizes of the particles in this material; i.e a wide size distribution.
3.6 Concluding Remarks

Different kinds of particle characterisations were carried out by using different methods: SEM, LD and IA. Also, compressibility, Zeta potential and cluster formation of all solid materials were investigated and tabulated under various conditions. From these results, it can be concluded that, both talc and calcium carbonate (calcite) have almost the same size distribution and Sauter mean diameter, while P25 is much smaller. Further, no evidence of particle cluster formation in talc and calcite, but clustering was clear in P25, when its suspension pH was increased from the natural pH= 2.5 to 5.3 (near to the IEP). Particle shape coefficient of different solids was obtained using Heywood’s method of categorisation. The results show that, almost all solid materials fits into a region of less than 0.5 except spheres and cube particle shapes. Seven different categories of $F_{va}$ were determined in order to simplify further working with this method of shape characterisation.
CHAPTER FOUR

EXPERIMENTAL AND NUMERICAL METHODOLOGY

In this chapter, two different methods are explained. The first is the experimental method that is used to investigate the measurement analysis of various conditions. The second is using the CFD technique to develop an optimal solid-liquid porous media simulation model to assist in predicting permeability.

4.1 Solid Liquid Separation and Permeation Methodology

4.1.1 Permeation and Filtration Tests

Constant rate filtration (CRF) tests were performed in a clear acrylic cell with an inner diameter of 60 mm and total volume of 424 ml as represented in Figure 4.1, using a laboratory pump (peristaltic) to suck the filtrate through the filter medium and depositing cake (see Appendix-B). To ensure a clear filtrate in all tests and promote cake filtration, a metal microporous membrane, with 10 μm slots, manufactured by Micropore Technologies Ltd (Hatton, Derbyshire, UK) was used as the filter medium (for talc and calcium carbonate). This 10 μm rated membrane had a nominal thickness of 0.06 mm and a measured hydraulic permeability of $1.2 \times 10^{-13}$ m$^2$, when clean, tested by a series of water flow tests at different pressures.

When using P25 suspension and because of its fine particle size, filter paper (WhatmanTM 542) was used, which had a nominal pore size of 2.7 μm, and was fixed on top of a metal perforated plate. At the base of the cell, within the filtrate line, a pressure transducer (HCX Series Honeywell S&C) was used to monitor the vacuum. A vacuum gauge (WIKA Instruments Ltd) was also connected to the system in order to validate and check the sensor reading. The filtrate was pumped out using a peristaltic pump (Watson Marlow 401U/D1) and collected in a vessel placed on an electronic balance (OHAUS SPU601), see Appendix-B. Both, weight of the filtrate and pressure drop in the system, as a function of time, were recorded using a PC with Labview software as shown in Appendix-B. For the tests three different filtrate pump settings were used (10, 30 and 50%) with talc and calcium carbonate. However, with titanium dioxide four filtrate pump rates were used (10, 40, 80 and
99% of full-scale). This gave rise to different filtration rates and cake forming pressure profiles.

Figure 4.1: Schematic diagram of constant rate vacuum filtration equipment

The same suspension was reused many times, to avoid any variability in the feed material. The loss in the reused suspension was monitored between filtrations and found to be very low (approx. 1% between runs). The ‘dead volume’ below the microporous membrane and within the filter tubes was filled with filtrate from a filtration of similar suspension before starting an experiment, in order to avoid missing filtration data at the very early stages of the experiment due to filling this dead volume. Before each series of tests, the suspension was prepared and left at room temperature (20 ± 2°C) for at least 24 hrs, with periodic mixing. This ensured the system had reached ionic equilibrium and avoided thermal gradients. To validate the calculations of the final cake concentrations, a vernier calliper was used; measuring cake height over five different positions (see Figure 4.2) per test: each position equally spaced on a pitch circle diameter of 25 mm with another position at the centre, and using the
average cake height in a mass balance to give the cake concentration. Measurements were taken from the top of the cell to the filter medium before starting the experiment and to the cake surface at the end. The cake height was, therefore, the difference between these two. It was possible to clean the metal slotted filter medium after each run and reuse it.

For comparison with the CRF data, experimental tests were also performed in a similar filtration cell under conditions of constant pressure (CPF) that has already been presented (Mahdi 2008). The same material was filtered, using the same filter medium as before.

![Figure 4.2: The five measuring points of the cake height](image)

In the case of CPF, the vacuum was applied using a vacuum pump and the vacuum receiver was positioned on a scale balance in order to measure the mass of filtrate recovered with respect to filtration time. A needle valve was used to bleed air into the vacuum receiver and to enable different amounts of vacuum across the filter to be achieved. Permeation processes were also used in some cases (low initial concentration). In this process, to prevent filtration and to have better results on permeation, a valve (under the filtration cell) was closed, and the mixture was added to the filter cell for enough time to settle the particles before opening the valve and starting the experiment. Both filtrate (volume or weight) and total pressure in the system with time were recorded and plotted. All filtration constants in the general filtration equation are calculated from the slope and the intercept of plotting $\nu/V$ (in case of CPF) or $\Delta P$ (in case of CRF) against $V$ (see Eqs. (2.4.8) and (2.4.9) respectively).
4.1.2 Sedimentation Test

Sedimentation tests were carried out in glass vessel having an internal diameter equal to 160 mm. Such diameter was identified as being the best size after a preliminary investigation using a wide range of vessels sizes (DiGiovanni 2013). Solid and liquid were weighed in order to find the volume of each one using their densities and then the concentrations of the mixture were calculated. All the suspensions, at different solid concentrations, were always reused and maintained at a constant temperature (20 ±2°C). This was to ensure the system had reached ionic equilibrium and to avoid any thermal gradients, which is fundamentally important for obtaining reproducible solid settling behaviour. Both time and height of interface were recorded. After that, the height of interface was plotted against time in order to find the particle settling velocity (the gradient) in a liquid under gravity force at different concentrations. Using a force balance Eq. (4.1.1) for a non-compressing system, the permeability of each concentration can be deduced.

\[ k = \frac{\mu}{Cg(\rho_s - \rho)}u_s \]  

(4.1.1)

where, \( u_s \) is the setting velocity [m s\(^{-1}\)], \( \mu \) is the liquid viscosity [Pa s], \( \rho \) is the liquid density [kg m\(^{-3}\)], \( \rho_s \) is the solid density [kg m\(^{-3}\)], \( C \) is the solid concentration (v/v) and \( g \) is the acceleration due to gravity [m s\(^{-2}\)].

4.1.3 Filtration Data Analysis

Figure 4.3 demonstrates the general steps in calculating the average cake filtration resistance (\( a \)). Data for collected filtrate volume against total pressure was plotted and a 2\(^{nd}\) order polynomial was fitted as shown in Eq. (4.1.2):

\[ aV^2 + bV + D = 0 \]  

(4.1.2)

where \( a, b \) are constant, \( V \) is the filtrate volume [m\(^3\)], the term \( D \) represents either time or pressure, depending on the intended empirical fit. This data-smoothing procedure was adopted to remove the normal random fluctuations in measurements and it was found necessary to apply different polynomial equations to different regions as discussed in more details in Chapter Five. The filtrate rate at any instance in time resulted from differentiating the polynomial equation for filtrate volume with time. In
the flow chart, step 5, both the intercept \( (D) \) and \( dv/dV \) was obtained by plotting the experiment data for applied pressure and filtrate mass, or volume, with filtration time.

\[(*) \] \( a, b \) are constant, \( V \) is the filtrate volume, the term ‘\( D \)’ represents either time \( (t) \) or pressure \( (\Delta P) \), depending on the intended empirical fit and \( \Delta P_c \) is the pressure over the cake. Initial data points (stage 1 in Figure 5.11), which are used to calculate \( R_m \), should not be included in the trendline overall the data except in the case of incompressible material

\[(**) \] A series runs at different pressure are required in order to calculate \( C_0 \) and \( m \)

Figure 4.3: The flow chart of calculation steps for CRF technique to determine specific resistance
4.2 Particles Shape Coefficient Methodology

Pictures of all materials were taken using different devices, which depends on the size of particles. The Photo-Microscope (PM) was used for particle sizes bigger than 20 µm, while Scanning Electron Microscopy (SEM) used for smaller sizes. Heywood’s steps of determining the shape coefficient of a particle, which is already discussed in the shape particle section, Chapter Two, were used in this work. The mean ($T_m$) and maximum ($T_x$) thickness have been found from the image of particles with measuring software (Image-J), which helps to calculate the 2D dimensions that are length ($L$) and Breadth ($B$). Figure 4.4 shows the way of measuring the mean and the maximum thickness of the particle.

![Figure 4.4: Mean ($T_m$) and Maximum ($T_x$) Thickness of Particles](image)

As can be seen in Figure 4.4, for the measurement taken, mean thickness ($T_m$) for the sphere and the triangle particles can be found using Eq. (4.2.1):

$$T_m = \frac{T_1 + T_2 + ... + T_5 + ... T_n}{n}$$  \hspace{1cm} (4.2.1)

where $n$ is the number of measurements (lines) and maximum thickness ($T_x$) in this case is equal to $T_3$, while for the cube ($T_m = T_x$). The number of thickness measurements can be any value (not only the five illustrated), which depends on the size of particle and the quality of the picture. Some equipment and techniques such as Lasentec (FBRM) gives more than 200 values for each particle. In part B, where particles are irregular, $T_x$, which is represented by arrows, will be different to $T_m$, which is represented by lines, in all particles. In the case of a two dimensional (2D) picture where the thickness is not visible to measure, a ratio of particle length has
been used, which is from 0.1% for plate particles to 100% for cube particles (0.1, 1, 10, 25, 50, 75 and 100%).

Using this information and equations in the section (2.1.2.3) on particle shape (see Chapter Two), all variables ratio including flatness ratio ($m$) elongation ratio ($n$) area ratio ($r_a$) and prismatic ratio ($P_v$) can be found. The next step is the volume shape coefficient of particles, which can be calculated using these variables with Eq. (2.1.18). The results of shape coefficient were compared with the Heywood ones in Table 9.1 in Appendix-A. This comparison is used to determine in which class the particle fits. In order to summarise and illustrate the Heywood's method a flowchart was created. Figure 4.5 shows the main steps that are used to estimate the value of volume coefficient. Table 9.2 in Appendix-A provides values for Heywood's volumetric shape factor of different materials and particle shapes.

Figure 4.5: Flowchart of Heywood’s shape coefficient steps
4.3 The ANN model methodology

4.3.1 Input variables investigation and selection (numerically)

According to Naes (2002) introducing too many factors will result in a large network size and consequently increased processing time and decreased efficiency. Understanding the influence of input variables is of primary concern when developing a numerical model (Zargari et al. 2013). In order to overcome this difficulty, Principal Component Analysis (PCA) function was applied to the data and then the treated data was used as an input for the ANNs and NLR models, although, PCA is a built-in function for PCR and PLSR models. In this work, nine different variables were obtained and investigated numerically (using PCA) and experimentally (see Chapter Five).

Table 4.1 shows that, particle size is the most significant variable, having a correlation coefficient of 0.92. These results corroborate the theoretical models (e.g. K-C and H-B) on the importance of particle size on the porous media permeability. In the same table, the next important parameter is particle concentration though with much lower coefficient of correlation. Two other variables worthy of consideration are the shape and the size distribution of particles.

Table 4.1: The relationship between input and output variables (correlation work)

<table>
<thead>
<tr>
<th>Input Variables</th>
<th>Correlation with the Output</th>
</tr>
</thead>
<tbody>
<tr>
<td>$X_1$ Particle Size, $x_{sv}$</td>
<td>0.917</td>
</tr>
<tr>
<td>$X_2$ Particle Concentration (v/v)</td>
<td>0.195</td>
</tr>
<tr>
<td>$X_3$ Shape Coefficient, $F_{va}$</td>
<td>0.110</td>
</tr>
<tr>
<td>$X_4$ $x_{50}/x_{10}$</td>
<td>-0.056</td>
</tr>
</tbody>
</table>

- Average particle size (Sauter mean diameter, $x_{sv}$),
- Solid concentration, ($C$) by volume
- Particle shape coefficient ($F_{va}$) and
- Measure of spread ($x_{50}/x_{10}$)

The four variables mentioned above were identified as the effective variables in the prediction of permeability to be considered as the inputs of the numerical models. Two of these variables (particle size and concentration) are already explicitly presented in the theoretical models. The output is the measured permeability ($k$) that was obtained from experimental methods as presented in Table 7.1.
4.3.2 The ANN model design

In order to create a successful model, a number of investigations on different aspects is required e.g. the model design, the number of layers and the elements per layer, the connections between the layers and the transfer and training functions (Anderson et al. 1992). There are standard steps required for designing an ANN model to solve a problem as shown in Figure 4.6.

![Flowchart of ANN methodology](image)

**Figure 4.6: ANNs methodology flow chart**

Different variables (e.g. inputs number, transfer, learning and training functions) affect the ANN performance, but number of hidden layers and neurons are the most important. The number of hidden layers is based on the number of inputs as reported
in the literature. On the other hand, trial and error method is used to find the number of neurons in each hidden layer. The number of neurons in the output layer corresponds to the number of output variables that are desired (Saeedi et al. 2007, Zuluaga et al. 2002). Arpat et al. (1998) stated that, for reservoir permeability prediction, supervised algorithms are generally preferred. Using Feed-Forward Back-Propagation (FFP) network, which is a development technique, will ease several problems. Therefore, an ANN model was designed using the FFP network to determine the optimum number of layers and neurons.

To investigate the agreement with the analytical models such as K-C and H-B, different numerical models were studied. Some of them (ANN2, NLR2) consist of only two variables (as in the analytical models) and the others (ANN4, NLR4) use four variables (from the variable investigation) as shown later in Chapter Seven.
4.4 CFD Model Methodology

The CFD technique was used to investigate the phenomena of water flow through packed beds and to examine the effect of bed geometry and packing structure on the flow properties.

4.4.1 Boundary conditions

Boundary conditions are one important aspect in the CFD modelling, which is the starting point to design and implement the model. Some of these conditions are similar for different models and the rest vary from model to model depending on the required results. The boundary conditions used in this work are described below:

- The model is three-dimensional (3D).
- To limit the computational demands, in each case only one or two particles were used in a cell model.
- The fluid was defined as incompressible and the particles studied were assumed to be stationary.
- To model the unit cell (UC) as a representative and repeated piece of a packed bed. Periodic boundary conditions were used in order to have identical variables everywhere.
- For all the vertical faces of the cell, symmetry (slip) boundary condition was applied. However, no-slip boundary condition was implemented on all the impermeable walls including the test particle.
- The modified Reynolds number \((Re')\) is sufficiently low and was varied from 0.5 to 2 in order to minimise turbulent effects.
- The packed bed model was designed, built, meshed and implemented in the commercial Particle Tracing Module within COMSOL 4.3b.

4.4.2 Cell Design

In order to investigate the effect of particle position in the cell and also to find which is better to use in this work, three different UC models based on the particle position in the cell were tested. These models are: one central particle (A), eight eighths of particle on corners (B) and using both in one cell (C) as shown in Figure 4.7. This investigation will be discussed more in detail in Chapter Six.
Figure 4.7 Three different UCs design

Figure 4.8 illustrates the surface contour of pressure for the three investigated unit cells using CFD model.

Figure 4.8 The surface contour of pressure for the three UCs
EXPERIMENTAL AND NUMERICAL METHODOLOGY

As can be seen in Figure 4.8, by using one particle in the centre of a cell usually introduces a numerical error caused by the singularity where the sphere meets the inlet and subsequently the error ratio will be increased if the lowest voidage is used. However, this behaviour does not appear in the other two cell models (B and C). In order to find a better model to use as a comparison between the results obtained from the three different cell models (A, B and C) and the Happel-Brenner (H-B) model was made. This comparison was based on different particle size and bed voidage values as shown in Table 4.2.

### Table 4.2: The value of the used parameter

<table>
<thead>
<tr>
<th>No</th>
<th>Parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Size ( x_{sv} ) (microns)</td>
<td>1, 5, 10, 50 and 100</td>
</tr>
<tr>
<td>2</td>
<td>Voidage ((v/v))</td>
<td>1, 2, 3, and 4 times of (x_{sv})</td>
</tr>
<tr>
<td>3</td>
<td>Modified Reynolds number</td>
<td>0.5, 1, 1.5 and 2</td>
</tr>
</tbody>
</table>

The comparison results are presented in Figure 4.9, which explains that cell A starts with high error (19%) and then decreased to less than 4% because of the singularity discussed before. Also, cell C started with around 9% and decreased to approx. 7% error, while Cell B started with less than 1% and ends with 5% error. Cell B is the same cell model used by Happel and Brenner in their original analysis.

Figure 4.9 Comparison of the three UC models
The results show very good agreement between H-B and CFD models with the lowest voidage. One important reason for that is H-B is a solution to the Naver-Stokes equation for particle point contact. Therefore, the results of the lowest voidage possible (particle touching each other) are much closer with less error ratio in the case of using cell model B and C. Generally, both analytical and CFD permeability values are in an acceptable error range for all cases (less than 19%) as shown in Figure 4.9. However, using UC (B) showed the lowest error ratio (less than 1%) especially with the lowest voidage. As a result, cell B was selected to use for further investigation on permeability prediction of the porous media.

4.4.3 Mesh resolution study

It is known that the resolution of the computational mesh influences the accuracy of a numerical solution. Reducing the mesh size will reduce the difference between numerical and theoretical or experimental results. Also, decreasing the size of mesh elements leads to an increase in the number of mesh elements, which can substantially slow down the computational process and require high memory. Therefore, the optimal size of mesh elements needs to be determined. Figure 4.10 shows results for the mesh elements resolution investigation. In this study, the liquid flow rate was determined under different values of the mesh elements, which was refined until eventually the results are stabilised with no significant change. As a result, M2 was chosen as the optimal number of mesh elements in this case, see Figure 4.11.

Figure 4.10: The flow rate as a function of the mesh elements number
4.4.4 CFD permeability prediction method

Figure 4.12 demonstrates the steps in predicting permeability for solid-liquid porous medium using this CFD method. The CFD input is superficial velocity ($U_0$) which was found from a fixed modified Reynolds number ($Re'$), the specific surface area of particle ($S_v$) and bed voidage ($\varepsilon$) using the rearranged form of Eq. (2.5.3) as in Eq (4.4.1).

$$U_0 = \frac{S_v (1 - \varepsilon) \mu}{\rho} Re'.$$

(4.4.1)

Five different particle sizes and voidage values were investigated for each $Re'$ value as explained earlier in Table 4.2. In the CFD model, average velocity ($U_0$) was integrated at the inlet face to determine flow rate, which was used in the Darcy law Eq. (2.5.1) to predict permeability. In order to find the pressure gradient ($\Delta P'$), global evaluation of pressure on the Inlet face meeting the slip boundaries was performed and then divided by the cell length. For the cases of the lowest voidage (solid particle touches the cell wall) integration at the face was not used due to the numerical error involved in the singularity. The analytical permeability was calculated from the knowledge of voidage and the particle Sauter mean diameter ($x_{50}$) with the Happel-Brenner model Eq. (2.5.3). Both analytical (H-B) and simulated (CFD) permeability values were compared and the difference was observed to be very small: less than 10% in all cases.
4.4.5 Model validation:

It is essential to establish the accuracy of the model and to ensure the validity of the approach. The CFD model was validated using two methods: the first is predicting different flow rate by increasing the pressure gradient, i.e. a linear relationship as shown in Figure 4.13. There is a slight curve tending to the pressure side, which might be a sign of a very small turbulence around the particle.
The second method to validate this model is by comparing the CFD model results with analytical values using H-B model for different particle size and bed voidage values as shown in Figure 4.14.

![Figure 4.14: Comparison of permeability values using both CFD (dots) and H.B (line) models (CFD Validation)](image)

Normally, the total flow rate in the cell is constant, where the inlet flow rate equals to the outlet one (continuity). The results showed, there is no difference between using $Re'$ of 0.5 or 2, which means no turbulence effects were found. However, to be confident with the results $Re'$ of 0.5 will be used in all CFD model predictions.
4.5 Concluding Remarks

An appropriate method of shape coefficient measurement was explained in detail. Solid liquid separation and permeation processes were designed, validated and performed in order to obtain experimental data and then experimental relationships to be used in the mathematical and numerical models. A method was explained to calculate the specific cake resistance by measuring the total pressure difference applied across a filter medium and the cake. These values complemented the values of permeability taken from the open literature, for use in the artificial intelligence methods of correlating permeability with particle properties. Regarding the computer simulation work, three different cell models were designed and validated. Furthermore, boundary conditions, mesh and inputs were investigated for different conditions. The cell model B was selected and the final optimal values of mesh elements were chosen for use. In general, the CFD model showed an acceptable error, based on comparison with the H-B analytical model, of less than 10% in all cases. This work starts with the dynamics of single particles in order to develop an understanding of such behaviour of multi-particle systems.
CHAPTER FIVE

INPUT VARIABLES INVESTIGATION (EXPERIMENTALLY)

This chapter discusses the experimental investigation of the relationship between the input variables and the output of the permeability prediction system. Four input variables were found numerically to have a strong relationship with the output as explained in the previous chapter. The experimental work was performed due to two reasons. The first was to confirm the numerical investigation and selection of the inputs, and the second was to obtain reliable data that will be used later with the models for permeability prediction, in conjunction with whatever reliable data that could be found in the literature. Various cake filtration techniques were used to study the effect of different parameters on the measured permeability of the three solid materials studied. The materials have different particle characteristics such as size, shape and compressibility and their characterisation techniques are also discussed.

5.1 Cluster formation and particle size analysis

From Figure 3.1 it is not clear if these particles are loosely agglomerated, forming clusters, or composed of fused crystals. Fine particles that are loose may be dragged through the cake by the shear field into the lower regions of the filter cake and, are likely to be deposited at the filter medium. Another aspect that was considered is the ability of the particles to form clusters that may compress with a variable applied pressure, this has been shown to have a major influence on the analysis and modelling of filtration and sedimentation process, as discussed below. Hence, particle characterisation was performed to investigate the possibility of clustering.

The permeability of water through particulate solid structures, at different solid concentrations and final compactions, was investigated in the laboratory during: sedimentation using Eq. (4.1.1), permeation using Darcy law Eq. (2.5.1) and filtration using Eq. (2.4.11). The intention being to compare the experimental permeability to the well-known H-B cell model (Eq. (2.5.3)) taking into account possible particle clustering during the processes, and comparing the values with independently measured particle size data. In this work titanium dioxide (P25) was used because of its small size, which increases its ability to form clusters. Permeability values from
these processes were compared as shown in Figure 5.1. The solid compact concentration was determined by mass balance, based on the initial mass of solids used, and the final height of the compact formed. The uncertainty bars on the figure represent the minimum and maximum value that the concentration could possibly be, depending on the degree of precision of the measurement of the sediment, or cake height. This was measured to ±0.5 mm, hence the concentrations reflect values calculated from the mass balance with an uncertainty of ±0.5 mm around the measured height after the test.

Figure 5.1 shows three different zones of permeability values, based on the solid concentration are noticeable. In sedimentation, at low solid concentration the highest values of permeability were found as expected. It is reasonable that the liquid in this case easily flows over the large clusters. In permeation, permeability values were found lower than sedimentation and higher than filtration, due to the formation of a compact of intermediate solid concentration. In permeation with low applied pressure, fluid might flow around the surface of the cluster, while in filtration this phenomenon will not occur where the shear and applied pressure were significantly higher and there does not appear to be any evidence of clusters within the filter cake.

![Permeability vs Final Solid Concentration](image)

Figure 5.1: Dependence of permeability on final cake concentration in sedimentation, permeation and filtration tests.

As a result permeability decreases as suspension concentration increases and filtration occurs. The obtained permeability values from different tests were converted
to Sauter mean diameters ($x_{sv}$), using the rearranged form of H-B model Eq. (2.5.3) as shown in Eq. (2.5.3).

$$x_{sv} = \sqrt{\frac{12C(3+2C^{5/3})k}{(2-3C^{1/3}+3C^{5/3}-2C^2)}}$$  \hspace{1cm} (5.1.1)

were, $C$ is the cake concentration v/v and $k$ is the measured permeability [m$^2$].

These results are plotted in Figure 5.2, which shows that the sedimentation tests had cluster size of close to 9.4 $\mu$m but for the maximum shear filtration tests the particle size is slightly less than 100 nm. This information confirms the particle characterisation data presented in Figure 3.1c and Figure 3.4. The inference is that in high shear filtration the filtrate flows rapidly through the filter cake and can easily penetrate, or disrupt, cluster formation. Hence, the correct particle size to use for permeability prediction in H-B model Eq. (2.5.3) would be between the primary particle size (28 nm) or, possibly, the primary cluster size as determined by the LD tests using very high energy input (100 nm). It is possible to use the particle/cluster sizes determined in Figure 3.4 in a predictive manner for permeability as also illustrated in Figure 5.2, by the linear plots superimposed on this figure, described as follows.

The sedimentation data was performed at starting concentrations between 0.5 and 0.015 (v/v), and gave rise to a sediment bed of 0.03 (v/v). Using these two values of starting solid concentration, together with the low shear cluster size of 9.4 $\mu$m, the starting points for the two lines in the top right hand corner of the graph are determined. The ending point of both lines, in the bottom left hand corner of the graph, comes from the 28 nm determined by the surface area and density measurements. The explanation for using this value is that under high shear the fluid flow will be over the surface of the primary particles within the cake formed and not across any of the clusters. The precise cluster size to be used in the equation depends on the process itself i.e. for low shear the cluster size appears to be 9.4 $\mu$m in sedimentation and around 100 nm in higher shear permeation and filtration.
Hence, in the case of a material that forms clusters the measured permeability clearly varies with the cluster size that is formed within the process, which in turn is dependent on the prevailing shear in that process. This is shown in the results from Figure 5.2, relatively high concentration data, and in the particle size analysis data (see Figure 3.4) at low concentration. Thus, the presence of clusters has a major influence on the measured permeability during experiments and the occurrence of clusters makes particle characterisation difficult – where the data will be used for design purposes based on permeability. These difficulties go some way to explain the variation in data that can be seen in the literature when trying to correlate permeability with particle characterisation data. For the later numerical (ANN and MVR) modelling only data taken from the literature where these issues were not thought to have an influence on the results was used. This led to the rejection of a lot of published data for the ANN model development.
5.2 Particle Concentration (v/v)

Cake concentration was determined based on the volume of the starting concentration and the volume of filtered slurry, together with the measured filter cake heights and the filter area, using material balances. The relative standard deviation (RSD) of the height measurements is obtained from the average value divided by the standard deviation. The measured heights, calculated cake concentrations and RSD for calcium carbonate and talc are shown in Figure 5.3 and Figure 5.4, respectively, where the RSD of the cake concentration is determined from the variation in cake height measurements over the five data points measured. The results of the expected RSD, in general, are decreasing with increasing initial slurry concentration: from approx. ±12% (0.01 v/v) to less than ±4% (0.05 v/v) feed suspension for talc, an expected consequence of a greater cake height being formed. However, in case of using calcium carbonate the RSD varies: from approx. ±2.4% (0.09 v/v) to less than ±1% (0.21 v/v). It is assumed that the cake is of uniform concentration throughout its height. The uncertainty bars around the mean values in both figures represent RSD around the mean concentration value, based on the five height measurements used to calculate that concentration.

Figure 5.3 and Figure 5.4 show three different trends, which depend on pump speed and initial suspension concentration. Pump speed determines the filtration flow rate and, consequently, the pressure drop in the filter cake, which has the expected power-law type of relation with the cake concentration. The cake volume concentration was used to calculate the dry cake mass per unit volume of filtrate ($c$), Holdich (2002), using Eq. (2.4.10). One aspect of the reported work is to assess the applicability of the experimental technique to an industrially relevant filtration testing study. In such a case a slurry sample can be taken from a process stream, and the solids concentration by mass could be determined (by drying). However, obtaining a representative sample of the filter cake is more difficult and it is time-consuming to weigh and dry the entire cake.

The measurement of the cake height at the end of the experiment is a much more rapid method for determination of the dry cake mass per unit filtrate volume, and it can be used to check the uniformity of the cake concentration by comparison with the height predicted from a volume balance: i.e. $\text{cake volume} = \text{original volume} - \text{filtrate}$
volume. Using the knowledge of the filter area enables the cake height to be predicted and compared with the measured height. If the measured height is significantly higher than the predicted value, then the filter cake is not uniform. In the tests reported here the measured and predicted heights agreed to approximately 2%, suggesting that a uniform cake concentration can be assumed.

Figure 5.3: Effect of pressure on average cake concentration with initial concentrations 0.09 to 0.21 (v/v) of calcium carbonate, number in the figure indicates pump speed 5, 10 and 30, [error bars provide the RSD]

Figure 5.4: Effect of pressure on average cake concentration with initial concentrations 0.01 to 0.05 (v/v) of talc, number in the figure indicates pump speed 5, 10 and 30, [error bars provide the RSD]

Finally on the subject of using the experimental technique for the analysis of industrially relevant filtration testing using a sample taken from a process stream, if
confirmation of the cake uniformity by height measurement is not required, the simplest equation for dry cake mass per filtrate volume ($c$) at the end of the filtration can be calculated from the knowledge of the mass of slurry filtered ($M$) and the filtrate volume ($V_F$):

$$c = \frac{sM}{V_F}$$

(5.2.1)

where, $s$ is the solid concentration of the slurry to be filtered as a mass fraction. It is proposed that Eq. (5.2.1) would be used for most industrial slurries due to its simplicity and ability to be applied when filtering mixtures, where densities are less likely to be known.
5.3 Constant rate and constant pressure testing

5.3.1 Filtration conditions and benefits

According to literature including Greil et al. (1992), Tiller et al. (1960), Tiller (1955) and Tiller (1953) the advantages and disadvantages of constant pressure and constant rate filtration are:

*Constant pressure filtration*

Advantages

- A huge number of experimental studies.
- Since liquid flow in the cake is additive, the flow rate necessarily increases as the liquid approaches the medium compressing the filter cake.
- Simplicity of obtaining the data.

Disadvantages

- Calculations and results have to be treated with caution; there is often a lack of consistency, especially when different personnel perform the tests.
- At the start there may be experimental difficulties, such as establishing a constant pressure over the filter cake, due to a period of filter medium blocking.
- There is not enough time to study the initial filtration period

*Constant rate filtration*

Advantages

- Upper and lower limits on the variation of solids pressure in the filter cake can easily be obtained.
- Industry use is widespread (apart from vacuum filters).
- Constant growth rate of the cake is induced with reports of homogeneous particle incorporation into the growing cake surface.
- The particle packing structure is expected to be independent of cake thickness so that uniform compacts are formed.
Disadvantages

- The applied pressure has to be continuously increased with cake thickness.
- In case of investigating gas deliquoring under pressure then this test rig would need to be modified.

It is common in data analysis to assume that there is no effect of sedimentation during cake formation. Hence, the cake resistance and permeability are calculated using equations based on Darcy’s law for liquid flow through a cake from graphical plots of filtration pressure plotted against filtrate volume, or time (constant rate) or time over filtrate volume plotted against filtrate volume (constant pressure) as shown in Figure 5.5 and Figure 5.6 respectively. In general, during most filtration tests there are three regions during a test run, which are: cake formation, transition and flow through the already formed cake region (Couper et al. 2009, Li et al. 2005, Wakeman et al. 2005).

5.3.2 Specific Resistance and Permeability

Calcium carbonate was used in this work due to its ease of particle characterisation such as shape and compressibility. The two linear equations represented by Eqs. (2.4.9) and (2.4.8) represent the conventional method of analysis for determining specific resistance from constant rate and pressure filtrations respectively. Examples of the results from these tests are shown in Figure 5.5 and Figure 5.6 respectively. In Figure 5.5 three different starting concentrations are illustrated: 0.11, 0.15 and 0.21 (v/v). There is a slight intercept above the origin on the pressure axis, indicating a small but finite filter medium resistance, in accordance with Eq. (2.4.9). These three intercept values are very similar. The reason they are not the same value is because of the different initial concentrations were used and its effect on the fine particles immigrations, which results a slightly different medium resistance at the start. It is relatively easy to determine the intercept from the cut-away graph and linearity of these data plots after about 10 ml of filtrate and up to about 120 ml, after which the filtration is substantially completed.
Figure 5.5: Measured filtration pressure during filtration at three starting concentrations: 0.11, 0.15 and 0.21 (v/v) with 10 rpm pump speed.

For comparison, Figure 5.6 shows a typical set of data achieved using the constant pressure filtration equipment, at a constant feed concentration of 0.11 (v/v), and three different constant applied pressures: 0.2, 0.4 and 0.6 bar. Again the linearity is good but, as is typical of constant pressure filtration the intercept values are highly scattered and the trend with pressure (for a constant value of $R_m$) is not as would be expected from Eq. (2.4.8). It did not help to plot the initial data on an expanded scale, as it was very randomly scattered, as is often the case in constant pressure filtration.

Figure 5.6: Constant pressure filtration of calcite at 0.11 v/v starting concentration and three total applied pressures for analysis by parabolic rate law.

Both sets of data, together with a number of other tests, were used to determine the relation between specific resistance and pressure. The log plot for the constant rate
tests is shown in Figure 5.7, where the specific resistance and pressure values plotted were determined by the end point of the constant rate filtration data: i.e. the last recorded values for specific resistance with pressure using the rearranged form of Eq. (2.4.9) as illustrated in Eq. (5.3.1).

$$\alpha_{av} = \frac{A}{cV} \left[ \frac{\Delta PA}{\mu} \left( \frac{dt}{dV} \right) - R_m \right]$$

(5.3.1)

The end point of the filtration was deemed to be the most appropriate part of the filtration for this analysis, to ensure that the conditions within the filter cake had stabilised adequately; most specifically, the filter cake concentration and dry cake mass per unit volume of filtrate ($c$), (Rushton et al. 2000). Table 3.1 shows data comparing the filtration scale up constants obtained by using the two filtration techniques: constant rate and constant pressure. It is noticeable that the data does appear to be very similar, as would be expected for a material of moderate compressibility. Also included in Table 3.1 is the data for the cake concentration as a function of pressure, obtained from the data illustrated in Figure 5.3.

![Figure 5.7: Effect of pressure on average cake filtration resistance, initial concentrations from 0.09 to 0.21 (v/v) of calcium carbonate, numbers in the figure indicate pump speeds 5, 10 and 30 rpm, (CRF technique)](image)

When filtering compressible materials the dry cake mass per unit volume of filtrate ($c$) will vary with pressure, as concentration does, see Eq. (2.4.10). However, it is usual for the variation of concentration with pressure to be much less than the variation of specific resistance with pressure (Holdich 2002). Nevertheless, concentration will
vary during a constant rate filtration during the early stages, as the pressure applied rises from zero to its ultimate value. It is informative to investigate this further, by solving Eq. (5.3.1) for all times during the filtration and comparing the values of specific resistance, as a function of pressure, with the average values obtained at the end of all the filtrations; at known values of pressure and flow rate. Figure 5.7 provides all the data used to determine the specific resistance scale up constants, which were provided in Table 3.1. In all cases the specific resistance was determined from the last filtration data point: i.e. values of pressure and flow rate. As would be expected, a low pump speed provides a low pressure resulting in a lower specific resistance. A best fit line was used on the logarithmic data to provide the values for the scale-up constants, as indicated by the power law equation Eq. (3.3.1).

In Figure 5.8 the specific resistance at each time interval is calculated, according to Eq. (5.3.1), and this is compared to the specific resistance calculated using Eq. (3.3.1), where the cake forming pressure is used. In order to determine the cake forming pressure the filter medium resistance was assumed to be constant and equal to the value determined from Figure 5.5. This gave a value of $1.43 \times 10^9 \text{ m}^{-1}$. The agreement between the values of specific resistance calculated by Eqs. (3.3.1) and (5.3.1) is very good. Clearly, as an experimental technique it would be possible to use the constant rate filtration equipment to determine values for specific resistance as a function of pressure, provided the dry cake mass per unit volume of filtrate is recalculated at each time increment, as it was using Eq. (2.4.10) and then Eq. (5.3.1). Just one experiment would provide the data for specific resistance as a function of pressure over a wide range of pressures, but a few experiments are required to determine the cake concentration functionality with pressure as illustrated in Figure 5.3 (which provides a constitutive equation for the analysis used in Eq. (5.3.1)). In Figure 5.8, an additional comparison is provided by specific resistance calculated using the K-C model for permeability and hence specific resistance. The resulting equation is as shown in Eq. (5.3.2):

$$\alpha_{av} = \frac{36KC}{(1 - C)^2 x_{av} \rho_s} \quad (5.3.2)$$

where $K$ is the Kozeny constant, a value of 5 was used as conventionally assumed, and $x_{av}$ is the Sauter mean diameter of the particles.
It is fairly common for the ‘text book’ value of the Kozeny constant to provide values of permeability, and hence specific resistance, an order of magnitude lower resistance (higher permeability) than are measured. It is notable that this occurs even here when using very stable and well characterised solids, as evidenced by the data illustrated in Figure 3.3b. Hence, there is a significant need for laboratory testing when designing filtration systems and the constant rate system, described here, provides a convenient and thorough technique for such analysis. By changing the Kozeny constant to a value of 33 it was found possible to match the specific resistance data illustrated in Figure 5.8 but the ‘text book’ value of 5 is significantly in error.

The conventional technique for laboratory testing is a constant pressure filtration. Superficially, this has the attraction of providing a fixed and known pressure drop so that a limited number of experiments, typically four, may provide values of specific resistance and cake concentration as a function of pressure; to provide the data for Eq. (3.3.1) and its concentration equivalent equation.

![Graph](image)

Figure 5.8: The pressure effect during CRF test on the average specific cake resistance [0.21 v/v feed solids of calcium carbonate at 10 rpm] and theoretical resistance by K-C model.

However, the results are often very scattered and one reason for this is due to the lack of truly constant pressure conditions. Even if the total applied pressure remains constant, the pressure drop over the filter cake will vary during an experiment and hence so will the pressures that should be used in Eq. (3.3.1); where cake forming
and not total pressure should be used. At the start of a ‘constant’ pressure filtration the total pressure drop is over the filter medium and zero pressure is over the filter cake. During the initial stages the proportion of the pressure drop over the cake rises from zero to a finite amount. After a short while it is usually assumed that the total pressure drop is now acting over the cake, but between the start of the filtration, and the end, the pressure forming the cake will be changing. Hence, even during ‘constant’ pressure filtration the cake forming pressure is varying. The point of varying resistance over the filter medium is illustrated for the constant rate filtration data investigated here in Figure 5.9.

At the start the percentage of the total pressure drop due to the filter medium is 100%, but this quickly drops down to a negligible amount. The insert graph shows that, this is a period during which the applied pressure rises to 30 mbar, equal to 200 seconds. Hence, over the first 200 seconds the cake concentration would be expected to be changing rapidly. During constant pressure filtration this stabilisation period is likely to be faster, as the applied pressure is normally much higher, but there would also be a higher pressure driving the fine particles into the filtration medium. This will lead to a greater filtration medium resistance than when operating under the more mild pressures at the start of the filtration that constant rate uses.

![Figure 5.9: The percentage of total filtration resistance due to the filter medium as a function of total applied filtration pressure and time, (using CRF)](image)

To summarise, the constant rate laboratory equipment for analysis of filtration performance is best used as follows. A slurry sample to be tested is taken and
INPUT VARIABLES INVESTIGATION (EXPERIMENTALLY)

weighed \((M)\) and a separate small sample is weighed, dried and weighed again to provide a value for \('s'\). After filtration, the dry cake mass per unit volume of filtrate \((c)\) for the entire filtration can be determined from Eq. \((2.1.3)\). The average cake concentration by volume fraction \((C)\) can be determined from a rearranged form of Eq. \((2.4.10)\) as in Eq. \((5.3.3)\).

\[
C = \frac{1}{\rho_s(1 - s'/s\rho) - (\rho_s/c) + 1} \tag{5.3.3}
\]

At least three different cake forming pressure ranges (pump speeds) should be used, to obtain data similar to that illustrated in Figure 5.3 and suitable for determining the cake concentration constants with pressure as provided in Table 3.1.

The initial stages of the filtration are important for the determination of \(R_m\), as illustrated in Figure 5.5. This is then assumed to remain constant during filtration, allowing the pressure drop over the filter medium to be calculated and hence the cake forming pressure. The average dry cake mass per unit volume of filtrate \((c)\) at any instance in time can be determined from Eq. \((2.4.10)\), using the cake volume fraction \((C)\) calculated for the pressure drop over the cake only. The specific cake resistance \((a)\), at the cake forming pressure \((\Delta P_c)\), can be calculated using Eq. \((5.3.1)\), where the total pressure difference \((\Delta P_T)\) is used in this equation. It is then possible to use Eq. \((3.3.1)\) to correlate specific resistance with cake forming pressure.
5.4 Initial stages of filtration and cake compressibility

The common objective in many laboratory filtration experiments include determination of the filter cake permeability, or specific cake resistance to filtration, and how it varies with filtration pressure as well as information on the filter medium resistance, after it has stabilised (Tarabara et al. 2002, Tiller 1953). It has been proposed that a homogeneous constant growth rate of cake using a constant rate provides better packing structure of particles that is independent of cake thickness (Greil et al. 1992). In the initial stages of filtration, the filter medium has no cake and the measured pressure is due to the resistance of the medium with an initial deposit for a given flow rate. The pressure then rises, depending on the slurry concentration and other parameters in the system (Chi 2006, Rushton et al. 2000, Greil et al. 1992).

Reports by many authors, Wakeman (2007), Wakeman et al. (2005), Wakeman et al. (2003a), Yim et al. (2001), Wakeman et al. (1991), Kotlyarov (1976) and Carman (1938), indicate that the resistances by both the cake and the medium play roles in determining the magnitude of the fluid flow rate at different times of the filtration process. For the cake and the medium, the measured resistances are influenced by the migration of the fine particles, which increases the interactions between the particles and reduces fluid flow rates through them. When filtering suspensions with low initial concentrations, the ability of fine particles to migrate into the filter medium is greater. Therefore, during the initial stages of filtration the filter medium resistance will rapidly increase to what is assumed to be a constant value, owing to clogging by fine particles, but often orders of magnitude greater than the medium resistance in the absence of solids (Wakeman 2007, Wakeman et al. 2005, Wakeman et al. 2003a Yim et al. 2001, Wakeman et al. 1991). Tiller et al. (1995) and Tiller (1990) concluded that, at the start of a filtration the initial average specific cake resistance is small but the resistance of filter medium at this period is large.

The shear within the filter cake was sufficient to ensure that clustering is not significant, unlike during sedimentation, as discussed in this chapter section 5.1, where an investigation on cluster formation during particle characterisation for filtration processes and during the filtration process itself was made. Generally, in the case of characterising particles for filtration, it is to be recommended that the particles
are measured in the same ionic medium to be used in practice and that a series of size analyses are performed at increasing energy, or shear, input to investigate the likelihood of cluster (or aggregate) formation.

Although the cake filtration behaviour of both incompressible and compressible filter cakes is now more fully understood, there still remains many fundamental issues of difficulty including a cake structure changing. Also, sedimentation influences during the initial stage of filtration is another fundamental issue that needs to be understood. According to Rietema (1953a) and Rietema (1953b) the so-called retarded packing compressibility (RPC) can be found in both constant pressure and constant rate filtration with almost any kind of material (compressible and incompressible). This phenomenon occurs only after some time after the initial filtration stage and for a critical cake height. During that time two different upper and lower cake layers were formed. However, the upper layer thickness retains its original packing and porosity, the lower is much denser packing with increasing resistance. He also found that, in the case of observing this phenomenon during constant rate filtration, the rate of pressure increase suddenly jumps to a higher and constant value which is due to the specific resistance increase at the bottom of the cake. As a result, the measured pressure and porosity distribution as well as the filtration rate with cake thickness are affected by this phenomenon. Similar behaviour to RPC was noted by Fathi-Najafi et al. (1995), Chase et al. (1994) and Baird et al. (1967).

On the other hand, RPC phenomena has only limited support, there are a number of researchers including Murase et al. (1989), Shirato et al. (1985) and Shirato et al. (1971) who do not accept Rietema’s results and it still has not fitted into the existing and developing theories of cake filtration. They suggested that this behaviour was related to the presence of pin electrodes that did not allow normal compression to occur within the experimental equipment. Moreover, Tarleton et al. (1997) and Sorensen et al. (1995) claim that the maximum solid concentration of the formed cake may be reached away from the medium at a position into the cake. Further, in some cases there are temporary increases in filtrate flow for a short time, which is accompanied by a sudden increase in the turbidity of the filtrate, and then the filtration rate returned to the original level. Tarleton et al. (2001) concluded that, this behaviour is more likely to occur during the filtration of suspensions containing
structured or more loosely bound particles. Further, he puts forward three hypotheses to explain the abrupt structural changes in a filter cake:

- Over time, a portion of the cake may become eroded by the flowing filtrate making it more porous. Thus, enhancing preferential flow. This can occur when the eroded portion is surrounded by more rigid regions, which can resist impact of new solids from suspension. However, this channel may eventually close (collapse) because of solid weight above it.
- The provisional increase in filtration rate is due to localised restructuring of the cake, causing the closure of preferential flow channels by the ingress of solids being filtered.
- Irreversible reduction in filtration rate is caused due to the abrupt macroscopic changes in the structure of the cake. This leads to the collapse of a significant portion in the cake formation. These collapsed particles then arrange themselves to form a new cake with overall low permeability and high resistance is similar to Rietema (1953) explanation of RPC.

Recognising these aspects, it is not easy to obtain values of average permeability or specific resistance for all materials used in filtration. The permeability of a filter cake is related to its cake specific resistance by Eq. (2.4.11). Efforts to predict the permeability (or resistance) to filtration from the particle size data are notoriously inaccurate. Hence, the need for empiricism for the purpose of scale up and design, and the uncertainty in the analysis of filtration results due to the above reviewed phenomena. Well known models of permeability, and hence specific resistance, contain terms representing the specific surface area per unit volume of the particles (and thereby the particle size) and the solids volume fraction concentration \((C, v/v)\). In the case of the Kozeny-Carman (K-C) expression it may be argued that the shape of the particles is included via a sphericity term \((\psi)\), as shown in Eq. (2.4.12).

The permeability could be converted to specific resistance using Eq. (2.4.11), but attempts to predict specific resistance from particle size analysis data usually fail by at least one order of magnitude, with the resistance being greater than that predicted. Advances in best practice for obtaining the data for filtration understanding, and modelling, are overdue and the most appropriate technique for data acquisition is CRF, as shown in section 5.3, with particular attention paid to the initial stages of
filtration and analysis of factors such as RPC. From this analysis average permeability, or cake resistance, values can be deduced and compared with the permeability models, but the data and research to-date appears to suggest that it is unlikely that a single analytical model such as K-C model Eq. (2.1.12) will be valid to correlate permeability (or cake resistance) with particle properties. A study on the ability to obtain reliable data on cake permeability, or resistance, from experimental procedures to investigate an alternative numerical model for predicting permeability is justified. In this section, two different particulate materials are reported, one mildly compressible (talc) the other substantially incompressible (calcium carbonate), their particle size distributions are very similar, as determined by laser diffraction (LD); this enables some discussion on the predicted and measured permeabilities from known analytical expressions and the measured values.

Figure 5.10 illustrates three different starting suspension concentrations 0.01, 0.03 and 0.05 (v/v) by volume of talc using the fastest pump speed, 30 rpm. Overall, the figures appear to be reasonable; with the highest suspension of solids showing the highest required filtration pressure. There was no observable evidence of significant gas bubbles being formed in the filtrate line even at the highest differential pressure (vacuum) of 0.7 bar. However, the shape of the figure is slightly unexpected in that there is a small point of inflection near the origin followed by a rapid rise in pressure before the rate of pressure rise reduces to a smooth increase in the latter part of the experiments.

![Figure 5.10: Measured filtration pressure during filtration at three starting concentrations: 0.01, 0.03 and 0.05 v/v of talc with 30 rpm pump speed](image-url)
Figure 5.11 shows the filtrate and total applied pressure difference with time for the talc filtration at the lowest pump speed (cake forming pressure). The filtrate curve can be broken into two visible sections: rapid filtration rate is labelled (1) and a slower filtration stage labelled (2). Both the filtrate mass and pressure curves illustrate this trend. The same type of behaviour was found for all the filtrations with the different materials. The initial stage (1) is illustrated in greater detail in Figure 5.12 for both talc and calcium carbonate (calcite) filtrations, which shows an even more complex situation apparently occurs. There appears to be an initial period of filtrate accumulation with respect to time, followed by a ‘plateau region’ during which no filtrate is formed. In the case of the more compressible talc this region is longer than the less compressible calcite. The pressure data is somewhat ‘noisy’ in both cases, due partly to the use of a peristaltic pump to suck the filtrate through the system and the operation of the transducer at a low differential pressure.

![Diagram](image)

Figure 5.11: The two stages during talc filtration at 5 rpm pump speed, (a) 0.01 v/v and (b) 0.05 v/v feed suspension concentration
The calcium carbonate (calcite) data is more noisy, due to the much lower pressures being recorded when compared to the talc filtrations. Despite this experimental noise, there is no evidence of a pressure difference plateau corresponding to the filtrate plateau. Hence, it can be concluded that the pressure provided from the pump continues to rise, but the filtrate rate stops for a period of up to 20 seconds. Further effort is made to try and understand this period as it is important from the point of view of attempting to determine the medium resistance, which is used later to provide a value for pressure drop over the medium and hence cake forming pressure drop.

Two possible explanations can be suggested for the unexpected behaviour that was reported. The first is the influence of sedimentation during the initial filtration. This was studied for the lowest (0.09 v/v) and highest (0.21 v/v) initial concentration of calcium carbonate. The results show that the filtration rate is much greater than the particle settling velocity by approx. 4 to 13 times for the lowest and highest initial
concentration used in this work, respectively. It can be seen that, particle sedimentation has some contribution on the lowest concentration while, the effect is very small and it can be neglected with the highest concentration. The second explanation is the use of a peristaltic pump to suck the filtrate through the filter medium. This was investigated and the analysis is illustrated in Figure 5.13, which shows the pump speed setting and the actual rotation time during the course of the filtration.

![Figure 5.13: Investigation on the variation of pump speed and rotation time during the filtration of talc at initial concentration of 0.05 v/v using a pump speed of 5 rpm](image)

The pump is a ‘low pulsation’ peristaltic pump with four rollers, hence at a setting of 5 rpm the time period for a roller to pass over the tubing is 3 seconds; i.e. if filtrate discharge occurs only when a roller passes then discharge should be every 3 seconds. Hence, this does not explain the stoppage of filtrate for a period of 20 seconds. This is the lowest pump setting used and the ‘plateau effect’ was noticed for all the experiments. Initially, experiments were performed with the outlet tube on the discharge side of the pump discharging into air above a beaker on the weighing scales, and the stoppage could have been due to the intermittent drainage of that discharge tube. So, the experimental procedure was changed to start with water in the beaker and the discharge tube end submerged in that water to prevent any drainage occurring. This alteration made no difference to the observation of a ‘plateau region’. Hence, it was concluded that the observed behaviour is due to the nature of the solids and the filtration and not the experimental procedure or equipment.
The data illustrated in Figure 5.11 and Figure 5.12 suggests that the pressure drop over the filter medium is negligible: the pressure being close to zero at the start of the filtration. However, in order to assess the relevancy of filter medium resistance in the later calculation of pressure drop over the filter cake the initial stages of filtration were treated as follows to provide three different possible values of $R_m$. Firstly, it was assumed that $R_m$ is equal to zero (N=0). Secondly the value of $R_m$ was determined by applying Darcy’s law to the first data point measured, neglecting any resistance due to the filter cake (N=1). The third approach is to consider the last time before the ‘plateau region’ occurs and again determine $R_m$ by assuming that all the pressure drop is over the filter medium by applying Darcy’s law (N=2). In the case of the talc filtration at 0.01 v/v solids, Figure 5.12a, this would be at 60 seconds into the filtration. This provided three values for $R_m$ that could be used in the later analysis for pressure forming the filter cake. The true value of $R_m$ would, of course, be somewhere between these extremes. However, the membrane resistance ($R_m$) for pure water is $R_m = L_m/k_m = 6\times10^{-5}$ (m) / $1.2\times10^{-13}$ (m$^2$) = $5\times10^8$ m$^{-1}$.

The shape of the filtrate with time curve shown in Figure 5.11 during period (2) demonstrates a slightly reducing filtrate rate with respect to time, whereas the pressure with time curve is substantially linear. This is attributed to the filtrate pump, it can be seen in Figure 5.13 that the pump speed slightly decreases, the measured rotation time increases, during this period. Hence, the pump motor is not sufficiently powerful enough to provide truly constant rate against a varying pressure formed by the filtration. This does not invalidate the filtration analysis so long as the actual pressure and filtrate rate can be determined at any instance in time, and that it is possible to assume an average cake concentration and specific resistance to filtration is valid.

Figure 5.14 shows the filtrate rate with time, both the differential of the second order polynomial and the experimentally determined values are illustrated. The latter are calculated by a ‘central difference’ technique, whereby for the filtrate rate ($V$) at time $t$, the rate is determined by Eq. (5.4.1):

$$\frac{dV}{dt}_2 = \frac{V_3 - V_1}{t_3 - t_1}$$  \hspace{1cm} (5.4.1)
It can be seen that the polynomial approach appears to be an adequate data smoothing technique for the analysis and filtrate rate will be used based on it for the later analysis of pressure drop over the filter medium, cake and thereby permeability and specific resistance of the filter cake.

Figure 5.14: Filtration flow rate with time and pressure for talc using 5 rpm pump speed, (a) 0.01 v/v, and (b) 0.05 v/v [2nd polynomial equation, Eq. (4.1.2) (-) and central difference (○), Eq. (5.4.1)]

The variation of average filter cake specific resistance to filtration with time, and with pressure, is illustrated for two example filtrations in Figure 5.15: (a+b) for talc and (c+d) for calcite. The analysis followed the format provided in Figure 5.10, one key aspect being to determine the pressure drop over the filter medium to deduct it from the measured total pressure drop to provide that for the cake. This was achieved by considering the three different values of \( R_m \), hence three different values of cake forming pressure were used to determine the illustrated three different values of cake resistance for all the filtrations.
In general, it is demonstrated that the pressure drop over the medium is negligible as the cake resistance is the same with respect to time, regardless of the chosen value of $R_m$. This is less obvious for the talc resistance with pressure curve (Figure 5.15b), but is a reasonable conclusion after a pressure of 80 mbar has been reached. Thus, for the purpose of determining the constitutive relation between cake resistance and cake forming pressure (Eq. (3.3.1)) the data illustrated in Figure 5.15b from 80 mbar onwards can be used.

![Comparison between calculated cake resistances using $R_m=0$ (○), $R_m=\text{N1}$ (●) and $R_m=\text{N2}$ (△), (a+b) for talc at 0.05 v/v and (c+d) for calcium carbonate at 0.21 v/v both using a pump speed of 5](image)

Figure 5.15: Comparison between calculated cake resistances using $R_m=0$ (○), $R_m=\text{N1}$ (●) and $R_m=\text{N2}$ (△), (a+b) for talc at 0.05 v/v and (c+d) for calcium carbonate at 0.21 v/v both using a pump speed of 5.

Considering the voidage formed by the two different systems and the commonly used Kozeny approach to permeability (Eq. (2.4.13)) it is possible to see why empiricism is still required for filtration modelling and scale up in design. Talc produced cakes with a concentration of between 0.18 to 0.26 solids concentration; the calcite cakes were between 0.41 to 0.53 in solids content (see Table 6.1). The Sauter mean diameter of
the two different solids were almost identical and, according to Eq.(2.4.13), the permeability of the calcite cakes (9.2x10^{-12} - 2.4x10^{-12}, m^2) should be substantially lower than those for talc (7.9x10^{-12} - 2.7x10^{-12}, m^2) as it provided the much lower voidage porous media for filtrate flow. Hence, the resistance should be much higher, Eq. (2.4.11). Even allowing for a low value of sphericity for talc (0.22) compared to calcite (0.81, Allen (2003)), would not explain the (at least) an order of magnitude higher specific resistance displayed by the talc cakes (9.5x10^{-7} - 2.5x10^{-8}, m kg^{-1}) compared to the calcite (2.3x10^{-8} - 4.7x10^{-8}, m kg^{-1}) ones. There is clearly an additional effect enhancing the resistance to permeation in the talc cakes compared to the calcite ones that is not recognised in the Kozeny approach. This may be related to the shape of the particles or the enhanced contribution to resistance from the fine particles with the size distribution. This is the main reason for the current project on use of ANN and MVR to determine permeability values, where particle shape and contribution of fines to the overall resistance is being investigated.
5.5 Concluding Remarks

Three very different solid materials were studied experimentally to investigate the factors influencing their experimentally measured permeability, and to illuminate some of the problems that routinely occur when determining permeability from filtration experiments. The latter point is important as robust data is needed for the numerical modelling (MVR and ANN), and although a lot of data may exist in the literature it may not be reliably deduced from experimental measurements for some of the reasons covered in the above discussions. It was found that, the prediction of permeability for the purpose of solid-liquid separation and contacting of colloidal suspensions is complicated by the variability of both concentration and apparent particle size due to cluster formation. Clustering can be beneficial for improving sedimentation rates and filtration permeability, but it is difficult to predict and control within designs. In this work, the Sauter mean diameters arising from the analysis, at different shear rates, and the apparent cluster (or particle) size were compared. The apparent cluster was observed during the separation processes of water through particle/cluster beds via the measured permeability and the predicted permeability based on Happel’s cell model. The deduced cluster size during permeation and filtration was close to the value obtained for particle size by laser diffraction at high energy input (approximately 100 nm), but significantly higher than the determined primary particle size (28 nm). Using the above two sizes (28 and 100 nm) as upper and lower limits in Happel’s permeability model it was possible to determine an ‘operating envelope’ of permeability that matched the measured values for the sedimentation, permeation and filtration processes. Cluster formation is less relevant in permeation and filtration than sedimentation. Characterising particles to be used in separation processes, it is recommended that the particles are measured in the same ionic medium used in practice and that a series of size analyses are performed at increasing energy input to check for any clustering effects.

The determination of cake concentration based on the volume of starting concentration and filtered slurry together with the measured filter cake heights and the filter area was made. The results show that, RSD has an inverse relationship with the initial slurry concentration. Also, pump speed determines the filtration flow rate and, consequently, the pressure drop in the filter cake, which has the expected
power-law type of relation with the cake concentration. From literature it is found that, the acquisition of design data for cake filtration is normally obtained from laboratory investigations performed under conditions of CPF technique. However, most non vacuum industrial filtrations are under conditions that are closer to CRF, where the pressure drop over the filter cake will be rising significantly and the cake concentration will also increase with time. Using CRF reliable data points at the start of the filtration can be determined, providing a more reliable value for the filter medium resistance. The robustness of this data is superior to what is normally obtained from the alternative CPF data. However, it is worth noting that the most noticeable variation of specific resistance with pressure is found within the lower pressure range; as pressure increases, the change in resistance becomes less pronounced as would be expected of a power law equation such as that shown in Eq. (3.3.1). Hence, the most relevant region to investigate is the lower pressure one.

Two different solid materials (talc and calcium carbonate) with similar values of Sauter mean diameter were used to investigate the initial stage of filtration. The filtration data plots for both types of solids demonstrate two different stages of filtration with all various initial concentrations and flow rates, which shows discontinuities in pressure and filtrate. In the analysis of filtration data for the purpose of determining the functional relation between cake forming pressure and filter cake specific resistance the investigator must exercise considerable care. Another aspect of the detailed analysis of the initial period of filtration is the possible occurrence of Retarded Packing Compressibility (RPC), which is still a controversial topic in filtration literature. The data illustrated here, for both mainly incompressible (calcium carbonate) and mildly compressible (talc) cake filtrations indicated that such a concept does appear to exist; with a significant period of zero filtrate flow despite continued application of filtration pressure. It is suggested that the filter cake underwent a period of rearrangement at that point, with the expressed liquid from that rearrangement flowing upwards into the slurry rather than down through the previously formed filter cake and the filter medium. Hence, after that 'cake collapse' the overall resistance to filtration changed to a higher value. The use of CRF for laboratory filtration analysis provides a means whereby the initial stages of filtration can be studied in detail, with a gently increasing pressure applied to the filter cake.
Estimating permeability or cake resistance from known analytical expressions is known to be inaccurate. The expressions often significantly underestimate the resistance to filtration and this is might be because of the ‘enhanced’ resistance due to particle shape and the presence of finer particles within the distribution that can possibly migrate within the forming cake. Hence, the intention of this thesis is to provide an alternative approach to permeability modelling based on either regression analysis, or ANN, both of which require a substantial set of ‘input’ data for the modelling work. It is apparent that some of this may come from previously published work of others, but only after ensuring that the data appears to be robust. Some of the discussion within this chapter provides reasons why this data may not be so robust. The alternative is to generate the data as part of the project, and this chapter explains how this may be achieved, with some degree of robustness checking, but this approach is very time consuming and alternative ways to generate the input data will be attempted.
CHAPTER SIX

OBTAINING PERMEABILITY DATA

In this chapter, the permeability values of different materials were obtained from three main sources: the literature, experiments and CFD simulation and used for permeability prediction. The materials studied had a wide range of properties such as particle size, size distribution and shape (most of them are regular) as shown in Chapter Three. Several solid initial concentrations were prepared and used. The CFD simulation model was designed and used in order to generate a large amount of data in less time with an acceptable accuracy for use with the regression and ANN prediction models.

6.1 From Literature

Around 530 data points were obtained from previous studies, (Mahdi 2008, Tarleton et al. 1999, Tarleton 1999, Tarleton et al. 1997, Holdich et al. 1993). These studies were conducted to investigate the permeability of the porous media for different materials characterisation and process parameters. The effect of various variables such as particle size, shape and size distribution, different applied pressure and flow rate, pH and solid concentration on permeability were examined. The Sauter mean diameter and size distributions of the particles were calculated using Malvern 2000 and Horiba LA-920 equipment laser diffraction based devices. Morphology of all materials was studied in order to find the shape coefficient of the particles, which was calculated using Heywood’s approach as discussed in Chapter Four. The Multivolume Pycnometer 1305 was used to measure the density of the materials. The data input was limited to the above literature studies arising from Loughborough personnel due to uncertainties when attempting to obtain data from similar studies reported in the literature; for example it is not common for authors to provide sufficient information on the shape of the particles used to enable its use in the training data set used in this work.

In order to check and to have confidence in data, all of these data points were recalculated using Darcy’s law and compared to the results based on both K-C and
H-B models. The method of outliers was also used here as a pre-processing routine for the ANN work (see Chapter Seven).

6.2 From Experiments

Three different materials were used (talc, calcium carbonate and titanium dioxide). The constant rate filtration (CRF) technique was performed with talc, while both filtration techniques (CRF and CPF) were applied using calcium carbonate. However, sedimentation, permeation and both filtration techniques were carried out with titanium dioxide (P25). P25 was used in order to investigate the particle cluster effect of colloidal material on the calculated porous media permeability. The measured heights of the final cake, calculated cake concentrations and relative standard deviation (RSD) for talc and calcium carbonate are shown in Table 6.1(a, b), where the RSD of the cake concentration is determined from the variation in cake height measurements over the measured five data points.

Table 6.1(a, b): Test results for the different initial solid concentration suspensions and various pump suction

<table>
<thead>
<tr>
<th>Ci (v/v)</th>
<th>P.Sp. 5 rpm</th>
<th>P.Sp. 10 rpm</th>
<th>P.Sp. 30 rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C.H (mm)</td>
<td>C.C (v/v)</td>
<td>RSD** (%)</td>
</tr>
<tr>
<td>0.01</td>
<td>5.57 0.191 12.98 10.49</td>
<td>5.46 0.194 13.09 12.25</td>
<td>4.74 0.224 11.07 16.98</td>
</tr>
<tr>
<td>0.02</td>
<td>11.72 0.181 6.99 8.66</td>
<td>10.62 0.199 4.21 9.01</td>
<td>8.53 0.249 6.46 11.83</td>
</tr>
<tr>
<td>0.03</td>
<td>17.38 0.183 7.38 7.23</td>
<td>14.67 0.217 8.07 6.23</td>
<td>12.13 0.263 7.81 8.95</td>
</tr>
<tr>
<td>0.04</td>
<td>20.27 0.209 3.86 5.59</td>
<td>19.43 0.219 6.47 5.47</td>
<td>16.12 0.263 5.32 7.39</td>
</tr>
<tr>
<td>0.05</td>
<td>26.58 0.199 4.03 4.65</td>
<td>24.69 0.215 3.89 4.50</td>
<td>20.09 0.264 6.41 6.38</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Ci (v/v)</th>
<th>P.Sp. 5 rpm</th>
<th>P.Sp. 10 rpm</th>
<th>P.Sp. 30 rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C.H (mm)</td>
<td>C.C (v/v)</td>
<td>RSD** (%)</td>
</tr>
<tr>
<td>0.09</td>
<td>20.12 0.475 2.45 4.77</td>
<td>19.31 0.494 2.32 5.41</td>
<td>18.28 0.522 2.23 4.43</td>
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<tr>
<td>0.11</td>
<td>24.36 0.479 1.69 1.49</td>
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<td>19.85 0.588 1.38 3.25</td>
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<tr>
<td>0.13</td>
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<td>27.76 0.497 2.53 1.52</td>
<td>26.61 0.518 2.24 5.51</td>
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<td>0.15</td>
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<td>32.33 0.492 1.15 4.00</td>
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<td>35.48 0.506 1.37 4.16</td>
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<td>0.21</td>
<td>51.81 0.430 1.20 1.59</td>
<td>44.68 0.499 1.13 3.85</td>
<td>42.09 0.528 0.98 4.87</td>
</tr>
</tbody>
</table>

* (Ci) Initial concentration, (P.Sp.) Pump speed, (C.H) Cake height, (C.C) Final cake concentration, (a) Cake resistance from filtration equation, Eq. (5.3.1).

** Relative standard deviation (RSD) based on the five points cake height measurement variability for each test leading to uncertainty value for cake concentration.
However, in the case of using titanium dioxide, the three processes: sedimentation, permeation and filtration were all used. Permeability measured values were obtained using different initial solid concentrations. Over 33 runs (data points) were achieved under variable conditions. The measured permeability corresponding to the different solid concentrations for sedimentation is reported as shown in Table 6.2. Table 6.3 shows the tests results at low initial concentration (0.007, 0.01 and 0.015 v/v) and pressure (pump speed), where significant sedimentation is taking place, filtration tests were not possible and only permeation tests after sedimentation could be completed. Hence, filtration was used with higher solids concentration (0.02 and 0.03 v/v), where the cake readily formed and a constant pressure established (it was not possible to use constant rate) as shown in Table 6.4. In addition, for intermediate values of concentration and pressure both permeation as well as CRF tests was possible to achieve.

Table 6.2: Test results of different initial solid concentration suspensions from sedimentation operation (P25)

<table>
<thead>
<tr>
<th>Initial Cons. (v/v)</th>
<th>Measured permeability k (m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.005</td>
<td>7.53E-10</td>
</tr>
<tr>
<td>0.007</td>
<td>4.37E-10</td>
</tr>
<tr>
<td>0.010</td>
<td>2.25E-10</td>
</tr>
<tr>
<td>0.015</td>
<td>5.38E-11</td>
</tr>
</tbody>
</table>

Table 6.3: Test results of different initial solid concentration suspensions and various pressure from peremation operation (P25)

<table>
<thead>
<tr>
<th>Initial Cons. (v/v)</th>
<th>Total pressure (Bar)</th>
<th>Cake Cons. (v/v)</th>
<th>Measured permeability k (m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.007</td>
<td>0.067</td>
<td>0.085</td>
<td>7.91E-15</td>
</tr>
<tr>
<td></td>
<td>0.210</td>
<td>0.085</td>
<td>5.78E-15</td>
</tr>
<tr>
<td></td>
<td>0.481</td>
<td>0.085</td>
<td>3.81E-15</td>
</tr>
<tr>
<td></td>
<td>0.545</td>
<td>0.085</td>
<td>3.38E-15</td>
</tr>
<tr>
<td>0.01</td>
<td>0.095</td>
<td>0.085</td>
<td>7.61E-15</td>
</tr>
<tr>
<td></td>
<td>0.298</td>
<td>0.085</td>
<td>4.43E-15</td>
</tr>
<tr>
<td></td>
<td>0.508</td>
<td>0.085</td>
<td>3.15E-15</td>
</tr>
<tr>
<td></td>
<td>0.596</td>
<td>0.085</td>
<td>2.87E-15</td>
</tr>
<tr>
<td>0.015</td>
<td>0.136</td>
<td>0.079</td>
<td>6.51E-15</td>
</tr>
<tr>
<td></td>
<td>0.501</td>
<td>0.079</td>
<td>2.77E-15</td>
</tr>
<tr>
<td></td>
<td>0.532</td>
<td>0.079</td>
<td>2.38E-15</td>
</tr>
<tr>
<td></td>
<td>0.637</td>
<td>0.079</td>
<td>2.10E-15</td>
</tr>
</tbody>
</table>

In total, more than 90 data points were obtained experimentially using three different materials under various conditions. Up to this stage of the project, two ways of obtaining data have been investigated and discussed.
It was found that, there were some difficulties for both methods. For example, data reliability is one of the greatest problems when taking data from literature. It was time consuming and not very cost effective to perform a large amount of experimental filtration work. In order to overcome these difficulties and generate as much data in the available time, with an acceptable accuracy, CFD modelling was designed, validated and used.

Table 6.4: Test results of different initial solid concentration suspensions and various pressure from filtration operation (P25)

<table>
<thead>
<tr>
<th>Initial Cons. (v/v)</th>
<th>Total pressure (Bar)</th>
<th>Cake Cons. (v/v)</th>
<th>Cake Resis. (m kg$^{-1}$)</th>
<th>Measured permeability ($k$ $(m^2)$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.007</td>
<td>0.481</td>
<td>0.119</td>
<td>1.892E+12</td>
<td>1.10E-15</td>
</tr>
<tr>
<td></td>
<td>0.545</td>
<td>0.148</td>
<td>1.624E+12</td>
<td>1.03E-15</td>
</tr>
<tr>
<td>0.010</td>
<td>0.298</td>
<td>0.113</td>
<td>1.925E+12</td>
<td>1.14E-15</td>
</tr>
<tr>
<td></td>
<td>0.508</td>
<td>0.130</td>
<td>1.347E+12</td>
<td>1.41E-15</td>
</tr>
<tr>
<td></td>
<td>0.596</td>
<td>0.141</td>
<td>1.392E+12</td>
<td>1.26E-15</td>
</tr>
<tr>
<td>0.015</td>
<td>0.501</td>
<td>0.106</td>
<td>1.856E+12</td>
<td>1.26E-15</td>
</tr>
<tr>
<td></td>
<td>0.532</td>
<td>0.116</td>
<td>1.217E+12</td>
<td>1.76E-15</td>
</tr>
<tr>
<td></td>
<td>0.637</td>
<td>0.127</td>
<td>1.78E+12</td>
<td>1.09E-15</td>
</tr>
<tr>
<td>0.020</td>
<td>0.405</td>
<td>0.100</td>
<td>6.43E+11</td>
<td>3.86E-15</td>
</tr>
<tr>
<td></td>
<td>0.440</td>
<td>0.103</td>
<td>3.051E+12</td>
<td>7.90E-16</td>
</tr>
<tr>
<td></td>
<td>0.491</td>
<td>0.109</td>
<td>2.922E+12</td>
<td>7.75E-16</td>
</tr>
<tr>
<td></td>
<td>0.566</td>
<td>0.113</td>
<td>3.697E+12</td>
<td>5.93E-16</td>
</tr>
<tr>
<td>0.030</td>
<td>0.474</td>
<td>0.134</td>
<td>6.17E+12</td>
<td>3.00E-16</td>
</tr>
<tr>
<td></td>
<td>0.581</td>
<td>0.141</td>
<td>6.161E+12</td>
<td>2.85E-16</td>
</tr>
<tr>
<td></td>
<td>0.667</td>
<td>0.150</td>
<td>6.121E+12</td>
<td>2.71E-16</td>
</tr>
</tbody>
</table>

6.3 From CFD Model

It is known that, CFD is a logical alternative to experimental work. Hence, investigating the input variables and generating more data points for use with the permeability prediction models are the main two aspects for using the CFD technique in this project. The variables (size, concentration and shape of particle) were studied using a CFD model to find the effect of each on the porous media permeability results. One advantage of using CFD is that a uniform characteristic (size, shape and packed bed) of particles can be investigated in detail, which is not possible in real material.

At first, a sphere was investigated based in terms of size and voidage in order to simplify and develop the work. The Happel-Brenner (H-B) model was used to compare and validate the CFD results for spheres. The same CFD model was also applied after that with other particle shapes (non-spherical).
6.3.1 Particle Size

Five different Sauter mean diameters ($\bar{x}_{sv} = 1, 5, 10, 50$ and $100 \, \mu m$) were investigated using spherical particles. Figure 6.1 illustrates the geometry, velocity magnitude [m s$^{-1}$] and pressure [Pa] for the minimum and the maximum particle size (1 and $100 \, \mu m$). The results of the five particle sizes against permeability using both predicted models (CFD and H-B) are shown in Figure 6.2, which presents porous media permeability variation with particle size. The simulated permeability increased from $2.0 \times 10^{-15}$ to $2.5 \times 10^{-11} \, [m^2]$ by increasing the particle size from 1 to $100 \, [\mu m]$ respectively.

![Image of CFD model results for the lowest and highest size of particle (1 and 100 \, \mu m)]

Figure 6.1: The CFD model results for the lowest and highest size of particle (1 and 100 \, \mu m)
Figure 6.2: Comparison between particle size vs. permeability from analytical (H-B) and numerical (CFD) models (for the same particle shape and voidage)

6.3.2 Voidage (Porosity)

Voidage ($\varepsilon$) represents the unoccupied (fluid) part of the porous media, which is the opposite to the concentration (solid). In case of using a sphere the minimum voidage (close packing) is 0.476 which is low compared to the close packing of other particle shapes. Figure 6.3 shows the CFD results of a sphere for the lowest and highest voidage using the same size of particle (10 $\mu$m). The results show that, velocity decreased significantly from 16.63 to 3.38 x 10$^{-3}$ [m s$^{-1}$] when the bed voidage increased from 0.476 to 0.998 v/v.

The comparison between predicted permeability and bed voidage based on both models (CFD and H-B) shows a direct relationship as illustrated in Figure 6.4. Permeability depends on both parameters (particle size and voidage), which is also reflected in these results.
Figure 6.3: The CFD model results for the lowest and highest voidage (0.476 and 0.998), (for $x_{\text{sv}} = 10 \, \mu m$)

Figure 6.4: Permeability variation depending on voidage (for the same particle size and shape), (for $x_{\text{sv}} = 10 \, \mu m$)
6.3.3 Shape of particle

Shape of particle has an effect on the porous media permeability as was found numerically in Chapter Four. The analytical models (i.e. H-B) work well only when all the particles are assumed to be spheres. There is no doubt that the way the non-spherical particle is oriented will have an effect on the results. Three more shapes (cube, plate and dendritic) were studied using CFD in order to have a variety of shapes and to consider their influence on porous media permeability. The effect of particle shape on flow rate and pressure drop during a fluid flow was found and used with Darcy’s law to predict permeability. The Sauter mean diameter was used, in order to have the same size but different particle shapes. It is defined as the equivalent spherical diameter based on the surface area per unit volume. The surface area ($A_s$) and volume ($V$) of each particle were calculated depending on its shape and then both specific surface ($S_v$) and Sauter mean diameter ($x_{sv}$) were obtained using Eqs. (5.4.1) and (6.3.2).

\[
S_v = \frac{A_s}{V} \quad (6.3.1)
\]

\[
x_{sv} = \frac{6}{S_v} \quad (6.3.2)
\]

The CFD results for both a sphere and a cube for the smallest size (1 µm) and lowest voidage (close packed) are shown in Figure 6.5. As illustrated in Figure 6.6, the H-B model prediction agrees with the CFD prediction for spheres with less than 1% difference in this case. Also, the CFD prediction for cubic particles is lower than the H-B model by around one order of magnitude. Both cubic and spherical particles results are very close to each other. So the difference between both models (analytical and CFD) will not be high as shown in Figure 6.6. However, the results show flow over a channel, where the majority of liquid flows through the centre of the cell and little flows between particles.
Figure 6.5: The CFD results for sphere and cube particles

Figure 6.6: The variation of permeability vs. voidage for two different particle sizes of sphere and cube particles

In the case of plate like particles two orientations were studied: through the plane and in the plane, as illustrated in Figure 6.7. However, the plate dimensions will be: length
(L) equals to width (W) and thickness (T) equals to 0.1xL. Typically, plate particles have relatively low solid concentration and high voidage (from 0.8 to 0.9) and it is quite difficult to get a packed bed with more than 0.3 v/v concentration (Tarleton 1998). This kind of plate is similar to talc and clay.

Typically, plate particles have relatively low solid concentration and high voidage (from 0.8 to 0.9) and it is quite difficult to get a packed bed with more than 0.3 v/v concentration (Tarleton 1998).

Figure 6.7: The CFD results of the two different orientations of plate particle

The drag force of an object is calculated by integrating the component of the stress tensor parallel to the flow (x-component) over the object's surface, which is denoted "spf.T_stressx" in COMSOL. Dividing the overall drag force of the particle by its projected area gives the total shear stress (including pressure and turbulence) and it will be over a distance that is the pressure gradient (ΔP/L) for use with Darcy's law. The projected area of plate-V (vertical to the flow) is higher by 10 times than in plate-H (Horizontal with the flow) for the same particle size and shape. Theoretically, drag force for plate-H is higher than plate-V and porous permeability is vice versa. In both cases (H and V) flow rate is assumed to be constant and the velocity is much higher with V than H because of the available space to flow. Figure 6.8 shows an opposite relationship between permeability and particle orientation with high and low value of
particle size (1 and 100 \( \mu m \)). From this figure, it can be seen that, the porous media permeability of plate-H is around 2.2 times of plate-V.

Figure 6.8: Permeability variation vs. voidage for two different particle sizes of plated particle (H and V)

There is a logical reason why the apparent permeability would be lower in plate-V than in plate-H to do with the small distance between particles due to voidage and packed bed structure. This can be explained by the illustration in Figure 6.9. In this figure, for both arrangements (H and V) the fluid is flowing down channel, which is different to flow around the particle. Generally, in regular packing fluid flows in channels, which is shown in these results. The vertical profile shows significantly larger and proportionally greater regions of free flow through the porous bed see Figure 6.9. The majority of the flow will be in wide open channels and the flow between the plates was negligible. So, the plates themselves impart relatively little drag. Comparatively, the horizontal case has less open channels and the channels between the plates are very small. Hence, the viscous stress will contribute more here, and thus yielded greater drag. Also, in the direction of the flow, the gaps between successive objects is much smaller for the "vertical" case, so flow is more effectively excluded from this region. In the horizontal case, the flow can more readily re-converge behind one periodic object and then be deflected again by the next. This is why researchers used the model of eight particles on the corner and one in the middle (cell C) with different particle shapes and no-slip conditions.
A dendritic (star) particle was made using a cube with six truncated cones on its faces as presented on Figure 6.10. It was not easy to have a voidage less than 0.62 even after adjusting the particle angles to fit in the cell because their edges touching each other.

According to these results, the effect of shape on permeability is low, which matches the previous results using the MATLAB ANN correlation to find the relationship strength between permeability and shape (see Chapter Four).
Regarding particle size distribution, there were possibilities. One of them is to keep the same size of the corner particles and add a central particle with different size each time. However, adding this middle particle will change the bed structure to irregular packing. As a result, it was not easy to investigate the size distribution of particles because of the available time.
6.4 Concluding Remarks

Over 960 data points were obtained using different methods. Numbers of materials with various characterisations were used with different techniques of measuring and determining porous media permeability. A major part of the work was to check the reliability of the data, which explains why experimental equipment and simulation modelling were designed and applied to the project.

Most of the analytical models for predicting permeability are not shape dependent (only spherical), while the CFD model should predict the permeability of any shape of particles with high accuracy. Four particle shapes were used including: sphere, cube, plate (V, H) and dendritic (star). It is found that, permeability strongly depends on particle size and voidage. Shape gives rise to form drag and it would be expected to have a significant influence on the resulting apparent permeability. The results did not show a clear effect within a porous medium. Spheres showed a good permeability prediction even with the lowest voidage. Also in the CFD work, the shape range was covered well using four different shapes. Surface area per unit volume ($S_v$) was used with non-spherical particles in order to avoid any difference in the calculation of equivalent sphere diameter ($x_{sv}$).

It would be easy to generate data from CFD models for different sizes, voidage and shape of particles and to a more limited extent particle size distribution. However, the CFD results presented was well correlated with the ANN correlation work, which suggests that shape and size distribution are not so significant in the determination of porous media permeability. Further work on CFD modelling might be relevant, but it is difficult to see how the effect of ‘migration of fines’ can be included within a CFD model for permeability.
CHAPTER SEVEN
COMPUTING PERMEABILITY

The need to develop a new approach that improves on existing permeability prediction is desirable. This is important so as to overcome the limitations of the analytical models discussed in Chapter Two of this thesis. Typically, there exists a number of ways that can be used to model permeability, however, this chapter will only highlight two techniques for model prediction: the use of Artificial Neural Network (ANN) and Multivariate Regression (MVR). Three different MVR models were used for prediction. These models include the Principal Component Regression (PCR), Partial Least Squares Regression (PLSR) and simple Nonlinear Regression (NLR). In addition, in order to improve the model training efficiency and prediction, pre and post processing routines, including PCA function, were applied on the data before feeding it to the model. This study demonstrates the use of neural-network predictors in conjunction with statistical techniques (MVR) to develop a model for predicting the permeability of a porous medium (and hence the flow rate). Data were obtained from various materials and processes under different conditions as discussed in the previous chapter. Parameters for the materials are: the size, concentration, shape and size distribution of particles. The processes used to obtain the data were filtration (constant pressure and rate), permeation and sedimentation. To accomplish this work, MATLAB neural networking toolbox as well as Microsoft Excel were used for graphing and data analysis purposes. In the course of this chapter, various processes such as pre-processing of the data, investigation of training functions, determination of optimum hidden layer and neuron number as well as the final evaluation of the model are reported.

7.1 Data Pre-Processing

The main objectives of pre-processing data when applying ANN technique are: to remove outliers (noise) and to obtain a training data set that serves as the characteristic of the input and output data (see Appendix-C). Some outliers are the result of incorrect measurements and can be immediately rejected and removed from the set of data (Walpole et al. 2012, Devore 2011). In this work, raw data was
COMPUTING PERMEABILITY

checked and cleaned statistically by removing all data points that do not follow similar data pattern.

7.2 Data Analysis

The variables used in this study for permeability prediction covered a large range of data as shown in Table 7.1.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Min</th>
<th>Max</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>$X_1$ Particle Size ($x_o$)</td>
<td>0.245E-6</td>
<td>168.0E-6</td>
<td>m</td>
</tr>
<tr>
<td>$X_2$ Particle Concentration, C (v/v)</td>
<td>0.015</td>
<td>0.628</td>
<td>-</td>
</tr>
<tr>
<td>$X_3$ Shape Coeff. ($F_{va}$)</td>
<td>0.0055</td>
<td>0.700</td>
<td>-</td>
</tr>
<tr>
<td>$X_4$ $x_{50}/x_{10}$</td>
<td>1.150</td>
<td>29.410</td>
<td>-</td>
</tr>
<tr>
<td>$k$ Measured Permeability</td>
<td>1.63E-17</td>
<td>5.3E-9</td>
<td>m$^2$</td>
</tr>
<tr>
<td>$k^*$ Output</td>
<td>-0.788</td>
<td>7.724</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.1: Distribution of the input and output variables

Due to this extensive data collection, the sets of data were treated using the Principal Component Analysis (PCA) function for easy and quick conversion. The main statistical descriptions of the data used are illustrated in Figure 7.1.

![Graphs showing distribution of input variables](image)

Figure 7.1: Distribution of the four input variables

About 90% of the particle size values are less than 10 µm and almost 75% of the solid concentrations are between 0.3 and 0.5 (v/v). Additionally, more than 90% of the particle size distribution values are less than 5 and more than 70% of the shape coefficient values are higher than 0.5. The preponderance of data at the fine particle
size is deliberate and useful. In the case of predicting permeability for design purposes, it is the prediction of finer particles permeability that poses the design problem, not that of larger particles.

### 7.3 ANN design and training results

In all cases, the ANN was implemented using the ANN module contained within the MATLAB environment. Initially, two different numbers of hidden layers (1 and 2) were studied to find the configuration that optimises the relationship between input and output. It was found that ANN model with one hidden layer suffices for this purpose. The different numbers of neurons in the hidden layers were investigated with ANN2 and ANN4 as shown in Table 7.2 and Table 7.3. The ANN code and design steps are shown in Appendix-C.

Table 7.2: An investigation on the number of input variables and neurons using TANSIG (transfer), TRAINBR (training) and LEARNGDM (learning) functions for 2 inputs (ANN model)

<table>
<thead>
<tr>
<th>X1</th>
<th>X2</th>
<th>X3</th>
<th>X4</th>
<th>Neuron</th>
<th>R² (Train)</th>
<th>R² (Test)</th>
<th>RMSE (Test)</th>
</tr>
</thead>
<tbody>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>0.897</td>
<td>0.938</td>
<td>0.170</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>0.945</td>
<td>0.955</td>
<td>0.126</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>0.954</td>
<td>0.948</td>
<td>0.120</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>4</td>
<td>0.960</td>
<td>0.979</td>
<td>0.107</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>6</td>
<td>0.961</td>
<td>0.980</td>
<td>0.104</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>0.964</td>
<td>0.981</td>
<td>0.118</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>[1 1]</td>
<td>0.922</td>
<td>0.978</td>
<td>0.128</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>[2 2]</td>
<td>0.935</td>
<td>0.979</td>
<td>0.119</td>
</tr>
<tr>
<td>xsv</td>
<td>C</td>
<td>-</td>
<td>-</td>
<td>[4 2]</td>
<td>0.955</td>
<td>0.980</td>
<td>0.114</td>
</tr>
</tbody>
</table>

Table 7.3: An investigation on the number of input variables and neurons using TANSIG (transfer), TRAINBR (training) and LEARNGDM (learning) functions for 3 inputs (ANN model)

<table>
<thead>
<tr>
<th>X1</th>
<th>X2</th>
<th>X3</th>
<th>X4</th>
<th>Neuron</th>
<th>R² (Train)</th>
<th>R² (Test)</th>
<th>RMSE (Test)</th>
</tr>
</thead>
<tbody>
<tr>
<td>xsv</td>
<td>-</td>
<td>F_{wa}</td>
<td>-</td>
<td>4</td>
<td>0.935</td>
<td>0.949</td>
<td>0.121</td>
</tr>
<tr>
<td>xsv</td>
<td>-</td>
<td>x_{50}/x_{10}</td>
<td>-</td>
<td>4</td>
<td>0.925</td>
<td>0.913</td>
<td>0.157</td>
</tr>
</tbody>
</table>

\(X_1, X_2, X_3\) and \(X_4\): are the input variables, RMSE is the root mean square error, \(x_{sv}\) is the particle size (Sauter mean diameter), microns, \(C\) is the particle concentration (v/v), \(x_{50}/x_{10}\) is the particle size distribution and \(F_{wa}\) is the particle shape coefficient.

The ANN models with various transfer, training and learning functions were run (Appendix-C). The results showed that the combination of TANSIG (tangent sigmoid), TRAINBR (bayesian-regularization) and LEARNGDM (gradient descent
weight/momentum) functions yielded optimum results (see Appendix-C). The ANNs architecture of ANN4 model (with four inputs) includes one hidden and one output layer as shown in Figure 7.2. Subsequently, in the case of using only two inputs, particle size (which had the highest correlation value) was studied with shape coefficient and size distribution as well as with particle concentration in order to investigate the best two inputs for use with the models. As can be seen in Table 7.2 and Table 7.3, twenty networks were modelled for both ANN cases (ANN2 and ANN4). Accuracy was measured using two accepted parameters: the coefficient of determination ($R^2$) and the root mean square error (RMSE). The text in bold indicates the optimum model. These results are shown later in Figure 7.5.

Table 7.3: An investigation on the number of input variables and neurons using TANSIG (transfer), TRAINBR (training) and LEARNGDM (learning) functions for 4 inputs (ANN model)

<table>
<thead>
<tr>
<th>X1</th>
<th>X2</th>
<th>X3</th>
<th>X4</th>
<th>Neuron</th>
<th>$R^2$ (Train)</th>
<th>$R^2$ (Test)</th>
<th>RMSE (Test)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>1</td>
<td>0.924</td>
<td>0.917</td>
<td>0.148</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>3</td>
<td>0.981</td>
<td>0.978</td>
<td>0.093</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>4</td>
<td>0.988</td>
<td>0.979</td>
<td>0.076</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>5</td>
<td>0.989</td>
<td>0.983</td>
<td>0.065</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>7</td>
<td>0.989</td>
<td>0.985</td>
<td>0.067</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>11</td>
<td>0.987</td>
<td>0.986</td>
<td>0.066</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>[2 2]</td>
<td>0.912</td>
<td>0.903</td>
<td>0.167</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>[4 2]</td>
<td>0.925</td>
<td>0.955</td>
<td>0.116</td>
</tr>
<tr>
<td>$x_{sv}$</td>
<td>$C$</td>
<td>$F_\text{v/v}$</td>
<td>$x_{50}/x_{10}$</td>
<td>[4 4]</td>
<td>0.933</td>
<td>0.968</td>
<td>0.097</td>
</tr>
</tbody>
</table>

$X_1$, $X_2$, $X_3$ and $X_4$: are the input variables, RMSE is the root mean square error, $x_{sv}$ is the particle size (Sauter mean diameter), microns, $C$ is the particle concentration (v/v), $x_{50}/x_{10}$ is the particle size distribution and $F_\text{v/v}$ is the particle shape coefficient.

As shown in Table 7.2, RMSE decreased as neuron number is increased until six neurons were used in a single hidden layer configuration. The RSME values are 0.170 and 0.104 for one and six neurons, respectively. However, it slightly increased with ten neurons used in the single hidden layer. With two hidden layers, the RMSE decreased from 0.128 to 0.114 for [1 1], and [4 2], respectively. The results showed that increasing the number of hidden layers from one to two did not decrease the RMSE value than using one hidden layer. In fact, the lowest RMSE values in the Table 7.2 are obtained with four and six neurons in the single hidden layer. Thus, the ANN models perform better with four and six neurons in a single hidden layer configuration. Since the difference between the RMSE values for the four and six
neurons is very negligible, it will be mathematically convenient to build practical models on few number of neurons e.g., four neurons in Table 7.2. Therefore, in this work, single hidden layer configuration with four neurons was selected for the case of two-input model (ANN2).

In Table 7.3, similar behaviours were observed for the RMSE values as the numbers of neurons and hidden layers change. In the table, single hidden layer configuration with five neurons performs best. Therefore, for the case of four-input model (ANN4), five neurons were used.

![Figure 7.2: The one hidden layer architecture for, [a] ANN2, four neurons and [b] ANN4 model, five neurons](image)

From all the information above, it can be seen that the selected ANN models give an acceptable performance based on both $R^2$ and RMSE. Furthermore, the ANN2 results in Table 7.2 are in good agreement with the analytical models when using only two variables (particle size and concentration) as inputs. The configuration of ANN models in this work is based on creating a Feed-Forward Back-Propagation (FFP) network with one hidden layer architecture using either 4 neurons (ANN2 model) or 5 neurons (ANN4 model), in the hidden layer (see Figure 7.2). Of all the architectures simulated, the ANN model with only one hidden layer and five nodes showed better results than the other models.
7.4 Predicted and measured permeability comparison

7.4.1 PCR, PLSR and NLR Models:

MVR based linear models can give a stable solution when using a larger number of principal components for slightly nonlinear data, while a nonlinear model can give better solutions when using fewer variables (Naes 2002). The prediction permeability function of the NLR model using the four input variables (NLR4) is shown in Eq. (7.4.1). This equation was found from a nonlinear regression package provided by XLSTAT, which is an Add-In application to Microsoft Excel®, (see Appendix-C)

\[
\begin{align*}
 k^* &= 3.922 + 0.406X_1 - 0.062X_2 - 0.419X_3 + 15.099X_4 \\
 &\quad - 0.912X_1^2 + 0.091X_2^2 + 0.496X_3^2 + 10.355X_4^2 \\
 &\quad + 0.357X_1^3 - 0.558X_2^3 + 0.513X_3^3 - 14.997X_4^3 \\
 &\quad + 0.308X_1^4 + 0.356X_2^4 - 0.238X_3^4 - 13.561X_4^4
\end{align*}
\]  

(7.4.1)

where \( k^* \) is the predicted output, \( X \) are the responses: \( X_1 \) is the Sauter mean diameter [m²], \( X_2 \) is the particle concentration (v/v), \( X_3 \) is the shape coefficient, \( X_4 \) is the particle spread (\( x_{50}/x_{10} \)). Both Eq. (7.4.2) and (7.4.3) are used to normalize (\( Y^* \)) and de-normalize (\( k^* \)) the data respectively.

\[
Y^* = \frac{2(X_i - X_{\text{max}})}{(X_{\text{max}} - X_{\text{min}})} - 1
\]  

(7.4.2)

\[
k^* = \left( \frac{k^* + 1}{2} \right) \left( k_{\text{max}}^* - k_{\text{min}}^* \right) + k_{\text{min}}^*
\]  

(7.4.3)

where \( X_i \) is the variable value, \( X_{\text{max}} \) and \( X_{\text{min}} \) are the maximum and minimum values of the inputs respectively that are shown in Table 7.1 for all inputs (\( X_1, X_2, X_3 \) and \( X_4 \)), \( k^* \) is the predicted output, \( k_{\text{max}}^* \) and \( k_{\text{min}}^* \) are the maximum and minimum values of the predicted output as in Table 7.1 respectively.

The Eq. (7.4.1) is used to predict the value of output (\( k^* \)) that is de-normalized using Eq. (7.4.3) before finding the anti-log10 and then multiplying with 1E-16, as demonstrated in the example of permeability prediction using NLR model shown in the Appendix-C. Figure 7.3 shows a flowchart describing this process. The RMSE and \( R^2 \) were used to quantify the prediction performances for PCR, PLSR and simple Nonlinear Regression (NLR) models. The values obtained for all of these models
(linear and nonlinear) using the 4 inputs (size, concentration, shape and size distribution) are shown in Table 7.4.

Table 7.4: Root Mean Square Error and Correlation Coefficient of prediction values for different MVR models used with four variables

<table>
<thead>
<tr>
<th></th>
<th>PCR</th>
<th>PLSR</th>
<th>NLR</th>
</tr>
</thead>
<tbody>
<tr>
<td>RMSE</td>
<td>0.553</td>
<td>0.556</td>
<td>0.0771</td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.496</td>
<td>0.490</td>
<td>0.962</td>
</tr>
</tbody>
</table>

As can be seen in Table 7.4 the performance criteria values for both PCR and PLSR models are similar, which could imply that these models give similar regression coefficients and prediction results as is often found in the literature. However, the NLR model shows much better prediction than both linear models as shown in Table 7.4 and Figure 7.4.

![Flow diagram of prediction steps of the NLR model](image)
7.4.2 ANN Model:

Figure 7.5 presents the predicted porous media permeability from both ANN models and shows that ANN4 predicts permeability better than ANN2. In addition, results of ANN4 and the analytical models (K-C and H-B) were compared with the measured (experimental) values (see Figure 7.6). This is carried out with the aim of...
understanding the relationship between them and to find the degree of difference. Measured results are represented by the dashed red line.

Figure 7.5: Predicted results of permeability using Artificial Neural network (ANN) model: (a) ANN2, using 2 inputs and (b) ANN4, using 4 inputs (---Represent the Measured data)

Figure 7.6 demonstrates that the ANN model results are closer to the measured results, with reduced error when compared to the analytical models. The average absolute error (AAE) was found with K-C and H-B models to be 35% and 40%, respectively. The results of using ANN2 model provide an error ratio of 14%.
However, the ANN4 model decreases the error ratio to approximately 9% compared to the measured results. One important reason for this reduced error is the addition of a shape coefficient and particle spread (fine ratio) in the ANN4 model. These two parameters are not inherent in the analytical relations, such as K-C and H-B models of permeability.

![Graph showing measured permeability values vs. ANN4 with two analytical models of permeability (K-C is Kozeny-Carman and H-B is Happel and Brenner).](image)

Figure 7.6: Measured permeability values vs. ANN4 with the two analytical models of permeability (K-C is Kozeny-Carman and H-B is Happel and Brenner)

A comparison of both nonlinear (NLR and ANN) models using RMSE and $R^2$ values under the same conditions (4 inputs), shows that the ANN model within MATLAB provides a better prediction, as presented in Figure 7.4 and Figure 7.5. However, the NLR model gives more advantages as the use of a special code is not required unlike in the case of MATLAB. In addition, the Excel software is easier to access and use compared to the MATLAB software. Furthermore, the AAE was calculated for all models. The results showed that, the AAE decreased significantly from about 40% to 9% by using the ANN4 with four inputs. Table 7.5 presents a summary of all the performance criteria for the models with their values. This table shows that, adding two more inputs improved the prediction of the nonlinear models.

In general, it can be observed that the ANN model in all cases successfully maps the training data and provides more accurate prediction values of permeability. The ANN model results give a reasonable view of the link between permeability using different
methods and different conditions of materials and thus provide good predictions that are better than the analytical and the MVR models.

Table 7.5: Values of different performance criteria for the models

<table>
<thead>
<tr>
<th>Model</th>
<th>Type of model</th>
<th>$R^2$</th>
<th>RMSE</th>
<th>AAE (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H-B</td>
<td>Analytical models</td>
<td>-</td>
<td>-</td>
<td>40.4</td>
</tr>
<tr>
<td>K-C</td>
<td>Analytical models</td>
<td>-</td>
<td>-</td>
<td>35.2</td>
</tr>
<tr>
<td>NLR2</td>
<td>Nonlinear models</td>
<td>0.901</td>
<td>0.121</td>
<td>25.5</td>
</tr>
<tr>
<td>ANN2</td>
<td>Nonlinear models with two inputs</td>
<td>0.980</td>
<td>0.075</td>
<td>21.6</td>
</tr>
<tr>
<td>NLR4</td>
<td>Nonlinear models</td>
<td>0.962</td>
<td>0.077</td>
<td>14.3</td>
</tr>
<tr>
<td>ANN4</td>
<td>Nonlinear models with four inputs</td>
<td>0.990</td>
<td>0.054</td>
<td>9.6</td>
</tr>
</tbody>
</table>
7.5 Concluding Remarks

The main aim of the work presented in this chapter was to investigate methods to predict the permeability of a loose porous medium encountered in Chemical Engineering using a statistical modelling approach. This approach has been tried before for petroleum Reservoir Engineering, but the input variables in that modelling were very different and not relevant to the loose packings such as filter cakes, column packings, etc. Three different methods were investigated: linear regression models (PCR and PLSR), nonlinear models (NLR and ANN) and analytical models (K-C and H-B). The data used were taken from both experimental tests and CFD simulation as well as what is reliably reported in literature of filter cakes permeability for different materials.

A statistical analysis of the data was done in order to remove outliers present in the data. Principal component analysis (PCA) was then carried out and factor loadings obtained to identify the main variables. Applying PCA and experimental investigation of the input variables resulted in the use of four input variables (three are inherent from the particle characterisation process): Sauter mean diameter, the particle size spread (i.e. effect of particle fines and called ‘fines ratio’), particle shape coefficient (as based on Heywood’s approach) and concentration of the particle bed.

Based on performance criteria values $R^2$, RMSE and AAE, the performance of different linear regression models was analysed and compared to nonlinear regression and ANN models. From this comparison, the ANN4 (four inputs) model showed better prediction than the other models. These ANN4 results were compared with the results of the analytical models: Kozeny-Carman (K-C) and Happel-Brenner (H-B) equations. Moreover, it was found that, using the ANN4 leads to an increase in the $R^2$ value from 0.90 to 0.99 and significant decrease in both the RMSE and the AAE values from 0.121 to 0.054 and from 40% to 9% respectively.

It can be concluded that the ANN model with four inputs and one hidden layer with five nodes provided the most reliable prediction results and achieved a better fit and forecast than the other models. It demonstrates that ANNs are capable of demonstrating sophisticated non-linear integrating effects. However, prediction of permeability using this ANN approach depends on the availability of the ANN code to
the user, it is based on MATLAB which is an industry standard, but it is not universally available and accessible. A simpler alternative approach is to use the NLR model within Excel, using the constitutive equations (Eq. (7.4.1)) provided in this thesis. This provides an accessible method for the prediction of permeability in non-consolidated porous media, for the purpose of modelling, based on the particle characterisation data from the particles contained in the media. This approach is summarised in the example provided in Appendix-C, where it is possible for an interested user to include the constitutive equations for permeability, from Eq. (7.4.1) to Eq. (7.4.3), in his or her spreadsheet and use them to predict permeability from the particle characterisation data. This approach is not as robust as the ANN model, but it is almost universally accessible and provides more reliable values of predicted permeability than the classic analytical models.
CHAPTER EIGHT

CONCLUSIONS AND RECOMMENDATIONS

8.1 Conclusions

Permeabilities of loose-porous media are of considerable interests in industrial processes. However, this important property can be seriously affected by the particle characteristics of the porous media. In this work, influences of particle characteristics on the permeabilities of different porous media have been thoroughly investigated via experiments, theoretical and numerical models with the aim of developing a more robust approach to the prediction of the permeability for different characteristics of loose-porous media under different conditions as encountered in Chemical Engineering processes.

Experimental investigation

Particle characterisations were conducted for talc, calcium carbonate and titanium dioxide (P25) using Scanning Electron Microscope (SEM), Laser Diffraction (LD) and Image Analysis (IA) to obtain the particle images, sizes, size distribution and shape respectively. Using Heywood’s method for measuring shape coefficient, it was found that almost all the material fits into a region of less than 0.5 except spheres and cube particle shapes. Based on the results, seven different categories of shape coefficient \( F_{vu} \) were determined. An investigation on cluster formation for the materials was made using the LD equipment. The results showed no evidence of particle cluster formation for talc and calcium carbonate (calcite) suspensions, but clustering was found in P25 titania suspension when the natural pH was increased from 2.5 to 5.3 (near to the IEP). Under these conditions, the reported particle size distribution by the LD depended strongly on the shear used within the analysis. This highlights the problem of obtaining reliable particle characterisation data, which is the essential starting point for any model of permeability.

Different solid-liquid separation and permeation processes were designed, validated and performed in order to obtain experimental data and relationships for use in the mathematical and numerical models. To select a more appropriate experimental technique for filtration, laboratory investigations were carried out on Constant Rate
Filtration (CRF) and Constant Pressure Filtration (CPF) techniques. Findings showed that the CRF technique, which is widespread in industrial use but not so within the laboratory, was more applicable for filtration as it started with zero pressure gradients unlike CPF, where the pressure drop over the filter cake changed significantly at the start of the filtration and the cake concentration also increased with time. Also, with CRF there was a gradual increase in the pressure resulting and a constant growth rate of the cake with more homogenous structure than when obtained with CPF. This prevents bleeding of fines towards the membrane, or filter septum, which might affect the calculation of the cake resistance. Thus, CRF was adopted as the method of choice in all investigations. Experiments were conducted to investigate the effects of the cluster formation, cake concentration, flow rate (pump speed) and the stages of filtration on the measured values of permeability.

Cluster formation (size and size distribution of particles) of P25 particles showed that the prediction of permeability for the purpose of solid-liquid separation and contacting of colloidal suspensions is complicated by the variability of both concentration and apparent particle size due to cluster formation. However, cluster formation is less relevant in permeation and filtration than sedimentation. Clustering can be beneficial for improving sedimentation rates and permeability, but it is difficult to predict and control within designs.

Relative standard deviation (RSD) of measured cake concentration decreases as the values of the initial suspension concentration increase. These were determined using two approaches. First from the volume of initial concentration (suspension) and filtered slurry and second from the measured filter cake heights and the filter area. Results from the two methods showed good agreement. Since the errors in the measured cake concentration gave an impact in the permeability values obtained, therefore, higher initial suspension concentration gives a more reliable permeability values.

Also, during CRF the pump speed determines the filtration flow rate and, consequently, the pressure drop in the filter cake, which has the expected power-law type of relation with the cake concentration. Thus, as the pump speed increases, the cake concentration increases as well and therefore results in a decrease in permeability.
CONCLUSIONS AND RECOMMENDATIONS

The filtration profile showed that the initial stage of filtration exists which depends on the material characteristics and process conditions. This stage was characterised by rapid filtration rate followed by a slower rate of filtration at the second stage. However, the average cake resistance determined at different stages of the filtration showed no dependence on this phenomenon. Thus, in all practical cases the measured value of average permeability is independent of the initial stage of the filtration.

**CFD model**

In addition to the experimental data obtained in the investigations described above, CFD was used to simulate permeability in packed beds in order to generate further robust permeability values under different conditions. The simulated data in combination with that of the experiments were later used for the numerical models (ANN and MVR) to predict permeability. Three different CFD cell models (A, B and C) were designed and validated. Furthermore, boundary conditions, mesh and inputs were investigated for different conditions. The cell model B, having eight eighths of particle on corners, was selected and the final optimal values were chosen. In general, the CFD model showed an acceptable error of less than 10% in all cases, based on comparison with the H-B analytical model under appropriate conditions (i.e. for a sphere).

Four particle shapes used were: sphere, cube, plate (V, H) and dendritic (star). The surface area per unit volume \((S_v)\) was used with non-spherical particles in order to avoid any difference in the calculation of equivalent sphere diameter \((x_{sv})\). It was found that, permeability strongly depends on the particle size and voidage. However, the particle shapes did not show any clear effect on permeability within a porous medium. Spheres showed good permeability prediction even with the lowest voidage (point contact) compared to the H-B cell model. However, there are no theoretical models based on non-spherical shapes to compare the results from the other three shapes. It would be easy to generate data from CFD models for different sizes, voidage and shape of particles and to a more limited extent particle size distribution.
Permeability modelling

To reach a definite conclusion on the most important parameters necessary in the modelling of porous media permeability, principal component analysis (PCA) was performed on over 530 data points obtained from experimental tests, CFD models as well as the literature. Prior to PCA analysis, statistical analysis was performed on over 960 data points and the outliers were identified and removed. The PCA function results showed that particle size is the most significant variable. This corroborates the theoretical models (e.g. K-C and H-B) as well as the findings from the CFD models and the experiments on the importance of particle size on the porous media permeability. Another important parameter is particle concentration with a much lower coefficient of correlation. Two other variables found important are the shape and the size distribution of particles. The four variables mentioned above were identified as the most effective variables in the prediction of permeability to be considered as the inputs of the numerical models.

The four variables identified in the above results were used to develop the permeability models. To do this, three different methods were investigated: linear regression models (PCR and PLSR), nonlinear models (NLR and ANN) and analytical models (K-C and H-B). The performance of the linear and nonlinear models was tested with $R^2$, RMSE and AAE while the analytical models were tested with AAE. It was found that the ANN with 4 input variables (ANN4) performed best compared to other models including analytical equations (K-C and H-B). Furthermore, the results confirm that the ANN model with four inputs and one hidden layer with five nodes (4-5-1) provide the most reliable prediction results and achieved a better fit and forecast than the other models. In addition, it was found that, using the ANN4 model leads to an increase in the $R^2$ from 0.90 to 0.99 and significant decrease in both the RMSE and the AAE from 0.121 to 0.054 and from 40 to 9% respectively.

Finally, the investigations and findings in this work demonstrate that relationships between permeability and the particle characteristics of the porous medium are highly nonlinear and complex. While the existing models have limitations in predicting this relationship, ANN shows a better prediction even with higher number of variables that are non-existent in the analytical models. This is owing to the ANN’s capability to
CONCLUSIONS AND RECOMMENDATIONS

catch sophisticated nonlinear integrating effects. However, using this ANN approach depends on the availability and accessibility of the ANN code to the user since it is based on MATLAB, which is an industry standard software. A simpler alternative approach is to use the NLR model within Excel, using the constitutive equations (Eq. (7.4.1)) provided in this work. This offers an accessible method for the prediction of permeability in non-consolidated porous media.
8.2 Recommendations and further work

Several recommendations emerge from this project that may help further development and applications in projects to determine particle permeability and their parameters. These recommendations are outlined as follows:

- To characterise particles for use in separation processes, it is recommended that the particles are measured in the same ionic medium used in practice.
- It is worth noting that the most noticeable variation of specific resistance with pressure is found within the lower pressure range. As pressure increases, the change in resistance becomes less pronounced as would be expected of a power law equation. Hence, the most relevant region to investigate is that of lower pressure where CRF provides more robust data.
- CRF technique is recommended for filtration laboratory experiments. This is because it provides a means whereby the initial stages of filtration can be studied in detail, with a gently increasing pressure applied to the filter cake.
- In the analysis of filtration data and for the purpose of determining the functional relation between cake forming pressure and filter cake specific resistance, the investigator must exercise considerable care regarding the onset of the initial stage of filtration.
- Further work on CFD modelling may be needed. The challenge is how the effect of ‘migration of fines’ can be included within a CFD model for permeability prediction.
- All experiments were carried out in water in this study. For future work, it may be valuable to work with different high viscous fluids such as silicone oil with different materials and various physical particle characteristics.
- Future work should investigate other packing arrangements such as regular, irregular and random ones.
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9.1 Appendix-A

9.1.1 Tables and Figures

Table 9.1: Measurement Statistical Values for $r_a$, $P_r$ and $C$ for Heywood’s four General Classes.
Source: Freshwater (1987)

<table>
<thead>
<tr>
<th>Shape group</th>
<th>$F_{ra}$</th>
<th>$C$</th>
<th>$r_a$</th>
<th>$P_r$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Geometrical forms:</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tetrahedral</td>
<td>0.328</td>
<td>4.36</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cubical</td>
<td>0.696</td>
<td>2.55</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Spherical</td>
<td>0.524</td>
<td>1.86</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Approximate forms:</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Angular: Tetrahedral</td>
<td>0.38</td>
<td>3.3</td>
<td>0.5 – 0.8</td>
<td>0.4 -0.53</td>
</tr>
<tr>
<td>: Prismoidal</td>
<td>0.47</td>
<td>3</td>
<td>0.5 - 0.9</td>
<td>0.53 -0.9</td>
</tr>
<tr>
<td>Sub-angular</td>
<td>0.51</td>
<td>2.6</td>
<td>0.65 - 0.85</td>
<td>0.55 -0.8</td>
</tr>
<tr>
<td>Rounded</td>
<td>0.54</td>
<td>2.1</td>
<td>0.73 - 0.82</td>
<td>0.62-0.75</td>
</tr>
</tbody>
</table>
Table 9.2: Values for Heywood's Volumetric Shape Coefficient

<table>
<thead>
<tr>
<th>No</th>
<th>Particle Type</th>
<th>Volumetric Shape Coefficient ($F_{va}$)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td><strong>Regular shapes:</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.</td>
<td>Sphere</td>
<td>0.524</td>
<td>a, b, c, d, e</td>
</tr>
<tr>
<td>2.</td>
<td>Cube</td>
<td>0.696</td>
<td>a, b, c, d, e</td>
</tr>
<tr>
<td>3.</td>
<td>Tetrahedron</td>
<td>0.328</td>
<td>a, b, c, d, e</td>
</tr>
<tr>
<td>4.</td>
<td>Cylinder with $E = 1$:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>viewed along axis</td>
<td>0.785</td>
<td>a, b, c, d, e</td>
</tr>
<tr>
<td></td>
<td>viewed normal to axis</td>
<td>0.547</td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>Spheroids: $E = 0.5$</td>
<td>0.262</td>
<td>a, b, c, d, e</td>
</tr>
<tr>
<td></td>
<td>$E = 2$</td>
<td>0.370</td>
<td></td>
</tr>
<tr>
<td>6.</td>
<td>Flat Tablet, $B=0.1L$</td>
<td>0.069</td>
<td>d</td>
</tr>
<tr>
<td>7.</td>
<td>Flat Tablet, $B=0.4L$</td>
<td>0.278</td>
<td>d</td>
</tr>
<tr>
<td>8.</td>
<td>Long Prism $L=5B$</td>
<td>0.311</td>
<td>d</td>
</tr>
<tr>
<td>9.</td>
<td>Long Prism $L=10B$</td>
<td>0.220</td>
<td>d</td>
</tr>
<tr>
<td>B</td>
<td><strong>Approximate values for isometric irregular shapes, A.C(H2):</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10.</td>
<td>Rounded</td>
<td>0.56</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>11.</td>
<td>Subangular</td>
<td>0.51</td>
<td>c, d, e</td>
</tr>
<tr>
<td>12.</td>
<td>Angular:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Tending to Prismatic</td>
<td>0.47</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td></td>
<td>Tending to a Tetrahedron</td>
<td>0.38</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td><strong>Selected natural particles (DI):</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>13.</td>
<td>Sand</td>
<td>0.26</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>14.</td>
<td>Sand (Rounded)</td>
<td>0.34</td>
<td>d</td>
</tr>
<tr>
<td>15.</td>
<td>Natural Coal Dust</td>
<td>0.20</td>
<td>d</td>
</tr>
<tr>
<td>16.</td>
<td>Bituminous Coal</td>
<td>0.23</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>17.</td>
<td>Pulverized Coal</td>
<td>0.25</td>
<td>d</td>
</tr>
<tr>
<td>18.</td>
<td>Blast Furnace Slag</td>
<td>0.19</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>19.</td>
<td>Limestone (Aragonite)</td>
<td>0.16</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>20.</td>
<td>Talc</td>
<td>0.16</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>21.</td>
<td>Gypsum</td>
<td>0.13</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>22.</td>
<td>Flake Graphite</td>
<td>0.023</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>23.</td>
<td>Mica</td>
<td>0.003</td>
<td>b, c, d, e</td>
</tr>
<tr>
<td>24.</td>
<td>Mica (Flakes)</td>
<td>0.03</td>
<td>d</td>
</tr>
<tr>
<td>25.</td>
<td>Fusain Fiber</td>
<td>0.10</td>
<td>d</td>
</tr>
<tr>
<td>26.</td>
<td>Glass (Angular)</td>
<td>0.28</td>
<td>d</td>
</tr>
<tr>
<td>27.</td>
<td>Flue Dust (Spherical)</td>
<td>0.41</td>
<td>d</td>
</tr>
<tr>
<td>28.</td>
<td>Flue Dust (Aggregates)</td>
<td>0.27</td>
<td>d</td>
</tr>
<tr>
<td>29.</td>
<td>Tungsten Powder</td>
<td>0.45</td>
<td>d</td>
</tr>
</tbody>
</table>
9.2 Appendix-B

9.2.1 The constant rate filtration system

The filtration cell information

Figure 9.1: The filtration cell (design and dimensions)

1- Suspension tank, height = 150 mm, D(in) = 60 mm and D(out)=70 mm
2- Seal (Gasket), D(in) = 60 mm and D(out)=69 mm
3- Metal microporous membrane, D(out)=69 mm
4- Membrane support, D(out)=69 mm
5- Cell base, D(in) = 70 mm and D(out)=90 mm
6- Clamping ring, D(in) = 70 mm and D(out)=90 mm
The microporous membrane:

Figure 9.2 illustrates a metal microporous membrane, with 10 $\mu$m slots, manufactured by Micropore Technologies Ltd (Hatton, Derbyshire, UK) was used. This 10 $\mu$m rated membrane had a nominal thickness of 0.06 mm and a measured hydraulic permeability of $1.2 \times 10^{-13}$ m$^2$, when clean, tested by a series of water flow tests at different pressures.

![Microporous membrane](image)

Figure 9.2: The metal microporous membrane used in this research

The pressure sensor:

A pressure transducer, HCX Series Honeywell S&C (HCX001D6V), was used to monitor the vacuum in the system (see Figure 9.3).

![Pressure transducer](image)

Figure 9.3: Pressure transducer (HCX Series)
**Pump**

The filtrate was pumped out using a peristaltic pump (Watson Marlow 401U/D1) that is shown in Figure 9.4.

![Figure 9.4: Watson Marlow 401U/D1 pump](image)

**Electronic balance**

The filtrate was collected in a vessel placed on an electronic balance (OHAUS SPU601), as shown in Figure 9.5.

![Figure 9.5: Electronic balance (OHAUS SPU601)](image)

**LabVIEW block diagram and interface**

National Instruments' LabVIEW software was used for data acquisition and recording the signal from acquisition device (NI USB-6009) which converted the analogue voltage into a digital signal as shown in Figure 9.6. The pressure sensor, the electronic balance and the pump were connected to this acquisition device in order to
monitor and record the pressure difference (mbar) and filtrate weight (g), as a function of time (s). Also, to control the speed of the pump as shown in Figure 9.7 and Figure 9.8. The pressure difference sensor was also connected to the base of the filtration cell, which was designed in the workshop of the Chemical Engineering Department at Loughborough University. The output signals were recorded digitally using LabVIEW software. Figure 9.7 shows a screenshot of the LabVIEW interface. Figure 9.8 presents the block diagram, which was built to record the signal as a text file for further analysis.

Figure 9.6: Data acquisition device, NI USB-6009

Figure 9.7: A screenshot of the LabVIEW interface
Figure 9.8: The LabVIEW block diagram
9.3 Appendix-C

9.3.1 Finding and excluding outliers data

Once the data was obtained, the next step is to calculate the boundaries for excluding outliers in the data set. Outliers can be a major source of skewness in any data set, which is assumed to be normal. Therefore, it is important that outliers are removed using a specific method e.g. distribution free, so it does not introduce possible bias into the analysis early on. This method is commonly referred to as the quartile or fourth-spread method. Essentially, the boundaries of each of the quartiles in the data set should be identified. After that, the fourth-spread ($S_f$, which is the distance between the lower and upper quartiles) was measured. Then, the upper and lower outlier boundaries as a function of $S_f$ were set. By creating a spreadsheet in the Excel file these steps can be made as follows:

- Find the minimum, median, maximum, 25th and 75th percentile boundaries of the data set for each column.
- Calculate the difference between the 75th and 25th percentiles that is the fourth-spread ($S_f$)
- The upper and lower outlier boundaries are equal to the median ±1.5 x $S_f$

At this point, outliers can be removed using Excel's built-in logic formulas to check the value of the data column, if it is above or below either one of the outlier boundaries. This formula says: If the value of the data in this cell is greater than the upper boundary or less than the lower boundary, then this cell is equal to 1, otherwise, 0.
9.3.2 Transfer Functions

The ANN includes many transfer functions that can be used with data (Khataee et al. 2010, Boroumand et al. 2005). Table 9.3 shows the most commonly used functions: the linear and sigmoid. In linear functions, neurons are used in the final layer of multilayer networks that are used as function approximations.

Table 9.3: Some Examples of the Transfer Function Icons.

<table>
<thead>
<tr>
<th>No</th>
<th>Function Name</th>
<th>Function Behaviour</th>
<th>Range</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Log-Sigmoid Transfer Function</td>
<td><img src="image" alt="Log-Sigmoid" /></td>
<td>$0 \leq a \leq 1$</td>
<td>$y_i^{(o)} = \frac{1}{1 + \exp(-x_i^{(o)})}$</td>
</tr>
<tr>
<td>2</td>
<td>Tan-Sigmoid Transfer Function</td>
<td><img src="image" alt="Tan-Sigmoid" /></td>
<td>$-1 \leq a \leq 1$</td>
<td>$Out = \tanh\left(\frac{Net}{2}\right)$</td>
</tr>
<tr>
<td>3</td>
<td>Linear Transfer Function</td>
<td><img src="image" alt="Linear" /></td>
<td>$-\infty \leq a \leq +\infty$</td>
<td>$Out = pureline\left(Net\right)$</td>
</tr>
</tbody>
</table>

An investigation on the transfer functions: TANSIG and LOGSIG functions using four inputs, where LINEAR function is recommended to use only within the output layer, was made. Table 9.4 shows that, TANSIG function give better prediction.

Table 9.4: ANN Transfer function investigation

<table>
<thead>
<tr>
<th>Function</th>
<th>Neuron Nu</th>
<th>R2</th>
<th>RMSE</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>TANSIG</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.969</td>
<td>0.091</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.979</td>
<td>0.078</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.983</td>
<td>0.058</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.978</td>
<td>0.079</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.975</td>
<td>0.083</td>
<td></td>
</tr>
<tr>
<td><strong>LOGSIG</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.963</td>
<td>0.097</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.964</td>
<td>0.096</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.973</td>
<td>0.085</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.955</td>
<td>0.10</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.951</td>
<td>0.12</td>
<td></td>
</tr>
</tbody>
</table>
In addition, the training and learning functions were studied as shown in Table 9.5 and Table 9.6 respectively.

### Table 9.5: ANN Training function investigation

<table>
<thead>
<tr>
<th>Functions</th>
<th>Type</th>
<th>Quality</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Trainbfg</td>
<td>BFGS quasi-Newton back-propagation</td>
<td>G. P.</td>
</tr>
<tr>
<td>2. Trainbr</td>
<td>Bayesian regularization</td>
<td>G. P.</td>
</tr>
<tr>
<td>3. Trainc</td>
<td>Cyclic order incremental update</td>
<td>V. P. P. and V. L. T.</td>
</tr>
<tr>
<td>4. Traingd</td>
<td>Gradient descent back-propagation</td>
<td>G. P.</td>
</tr>
<tr>
<td>5. Trainnda</td>
<td>Gradient descent with adaptive lr back-propagation</td>
<td>G. P.</td>
</tr>
<tr>
<td>6. Trainlm</td>
<td>Levenberg-Marquardt back-propagation</td>
<td>G. P.</td>
</tr>
<tr>
<td>7. Trainr</td>
<td>Random order incremental update</td>
<td>V. P. P. and V. L. T.</td>
</tr>
<tr>
<td>8. Trainscg</td>
<td>Scaled conjugate gradient back-propagation</td>
<td>V. P. P. and V. L. T.</td>
</tr>
</tbody>
</table>

### Table 9.6: ANN Learning function investigation

<table>
<thead>
<tr>
<th>Functions</th>
<th>Type</th>
<th>R2</th>
<th>RMSE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. learncom</td>
<td>Conscience bias learning function</td>
<td>not working</td>
<td></td>
</tr>
<tr>
<td>2. Learngd</td>
<td>Gradient descent weight/bias learning function</td>
<td>0.98</td>
<td>0.072</td>
</tr>
<tr>
<td>3. learnngdm</td>
<td>Gradient descent weight/momentum weight/bias learning function</td>
<td>0.99</td>
<td>0.054</td>
</tr>
<tr>
<td>4. Learnh</td>
<td>Hebb weight learning function</td>
<td>0.95*</td>
<td>0.116</td>
</tr>
<tr>
<td>5. Learnhd</td>
<td>Hebb with decay weight learning rule</td>
<td>0.98</td>
<td>0.071</td>
</tr>
<tr>
<td>6. Learnis</td>
<td>Instar weight learning function</td>
<td>0.98</td>
<td>0.074</td>
</tr>
<tr>
<td>7. Learnk</td>
<td>Kohonen weight learning function</td>
<td>0.99</td>
<td>0.072</td>
</tr>
<tr>
<td>8. Learnlv1</td>
<td>LVQ1 weight learning function</td>
<td>0.95</td>
<td>0.110</td>
</tr>
<tr>
<td>9. Learnlv2</td>
<td>LVQ2 weight learning function</td>
<td>0.48</td>
<td>0.650</td>
</tr>
<tr>
<td>10. Learnos</td>
<td>Outstar weight learning function</td>
<td>0.99</td>
<td>0.064</td>
</tr>
<tr>
<td>11. Learnp</td>
<td>Perceptron weight and bias learning function</td>
<td>0.99</td>
<td>0.066</td>
</tr>
<tr>
<td>12. Learnpn</td>
<td>Normalized perceptron weight and bias learning function</td>
<td>0.98</td>
<td>0.070</td>
</tr>
<tr>
<td>13. Learnsonm</td>
<td>Self-organizing map weight learning function</td>
<td>0.99</td>
<td>0.060</td>
</tr>
<tr>
<td>14. Learnwh</td>
<td>Widrow-Hoff weight and bias learning rule</td>
<td>not working</td>
<td>-</td>
</tr>
</tbody>
</table>

G.P. = Good Prediction, V. L. T. = Very Long Time, P. P. = Poor Prediction; *with trainlm
9.3.3 Investigation on the polynomial order

Table 9.7 shows an investigation on the polynomial equation using different order values. As a result, fourth (4\textsuperscript{th}) order gives the highest $R^2$ and lowest error values.

Table 9.7: Investigation on the order of the polynomial equation

<table>
<thead>
<tr>
<th>Order</th>
<th>$R^2$</th>
<th>RMSE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 2\textsuperscript{nd}</td>
<td>0.908</td>
<td>0.120</td>
</tr>
<tr>
<td>2. 3\textsuperscript{rd}</td>
<td>0.936</td>
<td>0.099</td>
</tr>
<tr>
<td>3. 4\textsuperscript{th}</td>
<td>0.962</td>
<td>0.077</td>
</tr>
<tr>
<td>4. 5\textsuperscript{th}</td>
<td>0.951</td>
<td>0.087</td>
</tr>
</tbody>
</table>
9.3.4 Example of permeability prediction using Regression

Using an Excel spreadsheet and following the steps discussed in the flow diagram (Figure 7.3), it can be found that:

Step (1):
- $X_1 =$ Sauter Mean Diameter : $4.827 \times 10^{-6}$ m
- $X_2 =$ Cake Concentration : $0.383$ v/v
- $X_3 =$ Shape Coeff.: $0.700$
- $X_4 =$ Size Distribution ($x_{50}/x_{10}$): $2.630$

Step (2): Normalize all values (inputs and the new output) using Eq.(7.4.2)
- $X_1 = -0.945$
- $X_2 = 0.202$
- $X_3 = 1.000$
- $X_4 = -0.895$

Step (3): Use Eq. (7.4.1) to predict the new output ($k^*$ = -0.102)

Step (4): De-normalize the predicted output ($k^*$) using Eq.(7.4.3) ($k' = 3.032$)

Step (5): Then Anti-log10 (3.032) = $1.076E+03$

Step (6): Finally, $k = 1.076E+03 \times 1E-16 = 1.076E-13$ m$^2$, which is the final predicted permeability for these input conditions.

For comparison, the measured permeability for this material was $1.052E-13$ m$^2$, so the difference between the measured and the predicted values is small ~2% using Eq. (2.6.8).
9.3.5 The ANN model code for predicting permeability

The following MATLAB code was implemented in this study to predict the permeability of porous media at different conditions of particle characteristics e.g. size, shape, particle spread and concentration of particles. This model code was divided to parts: training and testing new data. The steps can be expressed as follows:

- Reading all data (Input and output values) from Microsoft EXCEL spreadsheet file.
- Scaling (Normalize) the data to a mean of 0 and standard deviation of 1
- Creating feed-forward back-propagation (FFB) network with one hidden layer and five neurons in this layer
- Initializes the weights and biases of the network to improve results
- Trains the neural network
- Simulates the network, takes the network input ($p$) and the network object ($net$) and returns the network outputs ($y$)
- Converts the network output back into the original unit (De-normalize)
- Plot the ANN model results,
- Saving the network training results to reuse again

(MATLAB CODE)

```matlab
%% Permeability Modelling Using ANNs
%% Loughborough University, Chemical Dept.
%% By, Faiz M. Mahdi
%% Training and Testing Program

%% %---------------- Training Model (1/2)-----------------%

close all
clear all
clc
format short;

%% Reading Data

data=xlsread('PureData.xlsx');
```
inputs=data(1:461, 1:4)'; % all inputs
outputs=data(1:461, 7)'; % all outputs

% Normalize the Data
[u1, us] = mapminmax(inputs);
[y1, ys] = mapminmax(outputs);

% Create Neuron Network Model
net = newff(u1, y1, 4, {'tansig'}, 'trainbr', 'learngdm'); %
net.trainParam.epochs=1000;
net.trainParam.show=300;
net.trainParam.goal=1e-3;

% View the Network
% view(net) % view the network figure

% Set up Division of Data for Training, Validation and Testing
net.divideParam.trainRatio=80/100; % 90% of the data for Training
net.divideParam.valRatio=20/100; % 10% of the data for Validation
net.divideParam.testRatio=5/100; % 5% of the data for Testing

% Initialization of the Network
net=init(net); % initialize the weights and biases of the network to improve results

% Train the Network
[net,tr] = train(net, u1, y1);
X = getx(net);
plotperform(tr); grid on;
plottrainstate(tr); grid on;

% Apply (Simulate) the Network
ytrn = sim(net, u1);

% De-normalize the Data
outtrn_again=mapminmax('reverse', y1, ys);
ytrn_again=mapminmax('reverse', ytrn,ys);

% Plot the Model Results
plotregression(y1, ytrn); grid on;
plotregression(outtrn_again, ytrn_again); grid on;

figure(1);
[m1, b1, r1]=postreg(ytrn_again, outtrn_again);
% saveas(figure(1), 'figure_1.bmp');
% figure(2);  
% plot(msereg);  
% siveas (figure(2), 'figure_1.bmp');  
%% Generating Network Architecture (Simulink)  
%gensim(net); % present the diagram of the network  

%% Save the Network Training  
save Train_Perm1 % saving the network training results to reuse again to test new data  

%% ------------------------------- Testing Model (2/2)-------------------  
%load Train_Perm1  
%close all  

% Read the Testing Data  
intst=data(462:550, 1:4)'; % all inputs  
outttst=data(462:550, 7)'; % all outputs  

%% Normalize the Testing Data  
[u2, us2] = mapminmax(intst); % Normalize the test input and save the results in Y1  
[y2, ys2] = mapminmax(outtst); % Normalize the target and save the results in Y2  

%% Apply the Training Model  
ytst = sim(net, u2);  

%% De-normalize the Results  
intst_again=mapminmax('reverse', u2,us) ;  
ytst_again=mapminmax('reverse', ytst,ys);  
outtst_again=mapminmax('reverse', y2,ys);  

%% Plot the ANN Results  
figure(3);  
plot([ytst_again' outtst_again']); grid on;  
err = ytst_again-outtst_again;  
%saveas(figure(3), 'figure_2.bmp');  
%disp('Absolute error'); disp(err);  
%ploterrhist(err);  

figure(4);  
plot(err); grid on;  
title('Absolute error');  
relerr = abs((ytst_again-outtst_again))./outtst_again*100;  
%saveas(figure(4), 'figure_3.bmp');  
%disp('Relative error (%)='); disp(relerr);
figure(5);
plot(relerr); grid on;
title('Relative error (%)');
meanrelerr=mean(abs(relerr)); % average on each rows
%disp('mean of relative error (%)='); disp(meanrelerr);

mse=mse(err);
mse2=sum((ytst_again-outtst_again).^2)/16;
%saveas(figure(5),'figure_4.bmp');
disp('Test MSE='); disp(mse2);

figure(6);
[am,b,r]=postreg(ytst_again,outtst_again);
plotregression(outtst, ytst);grid on;
plotregression(outtst_again, ytst_again);grid on;
%saveas(figure(6), 'figure_5.bmp');

figure(7);
plot(ytst_again(1,:), '*b'); hold on;
plot(outtst_again(1,:), 'ok');grid on;
%saveas(figure(7),'figure_6.bmp');

title('Perm1', 'FontName', 'arial', 'color', 'k');
legend('simulated-test-Perm1', 'experimental-test-Perm1');
xlabel('Number of test data');
ylabel('Output of Perm1');

% Save the Final Results

save Test_Perm1
9.4 Appendix-E (List of Publications)

Journal Paper:

Published:


Submitted:


Manuscripts under Preparation:


Conference (Presentation):

   ➢ The third price student presentation
8. Abdul Shakoor and Faiz Mahdi, 2013, Influencing the filtration flow rate by coating the particulate substance, NPCEC-2013, RSC, Newcastle, UK, August 2013

10. **Faiz M. Mahdi**, 2011, Predicting Permeability using Artificial Neural Networks (ANNs), *Café Academique, Loughborough*, UK, **Nov. 2011**


**Conference (Poster):**


filtration, PSA 2011 Conference and Exhibition, RSC, Edinburgh, UK, Sep 2010

Conference (Abstract):

20. **Mahdi, F.M.** and Holdich, R.G., 2013. Particles characteristics effect on permeability prediction in porous media, *COMSOL Conference*, WTC Rotterdam, the Netherlands, **Oct. 2013**
