EXPERIMENTAL AND ANALYTICAL INVESTIGATIONS OF THE
RELATIVE AND INTERACTIVE EFFECTS OF PHYSICOCHEMICAL FLUID
CHARACTERISTICS ON THE INCIPIENT MOTION OF GRANULAR
PARTICLES UNDER LAMINAR FLOW CONDITIONS

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by
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ABSTRACT
This dissertation first presents the evolutionary development of an experimental methodology for studying the relative and interactive effects of three physicochemical fluid characteristics (viscosity, pH, and ionic strength) on the incipient motion of granular particles under laminar flow conditions. Critical flow velocity was used to quantify the relative erosive capacity of the test fluids and a major goal throughout the research was to decrease the variability in its experimental quantification. This goal guided four phases of evolutionary developments in the methodological and analytical design and implementation. A statistical design of experiments, the response surface methodology (RSM), was used to guide the design and implementation of 20 test fluids with various levels of the three fluid characteristics. Incipient motion of a highly spherical glass bead positioned atop a specially designed and manufactured support pocket within a flow cell was observed using a microscope video camera. The critical flow velocity was quantified for seven repeat trials for each of the 20 test fluids. Regression analyses were conducted on the results to generate a statistical model to describe the relative effects of the three factors and their interactions. An analytical study was then conducted to provide a theoretical explanation of the experimental observations. The main conclusions are as follows: (1) viscosity, pH, and their two-way interaction were determined to be the factors most influential on critical velocity and viscosity was shown to be of greater influence than pH; ionic strength was found to be of little impact; (2) viscosity and pH interacted such that when pH was higher (at 10.5) the viscosity had greater influence than when the pH was lower (at 3.5); (3) at a viscosity of approximately 0.007 g/cm·s, critical velocity remained relatively unchanged throughout the range of pH (3.5 to 10.5); (4) for
viscosities greater than 0.007 g/cm·s, critical velocity decreased with an increase in pH while for viscosities less than 0.007 g/cm·s, the effect of pH on critical velocity reversed; (5) at pH values from 3.5 to 10.5, critical velocity consistently decreased as viscosity increased; (6) both electrostatic and van der Waals forces are strongly dependent on the separation distance between two particles; (7) as pH increases and zeta potential decreases, the electrostatic force (repulsion) between a test particle and a bed of particles increases and the test particle is more likely to be mobilized, i.e., the critical flow velocity decreases; (8) as ionic strength increases, the repulsive electrostatic force generally decreases and could cause the net surface force on the test particle to change from repulsion to attraction. Thus, the test particle becomes less likely to be mobilized, i.e., critical flow velocity increases; (9) as zeta potential decreases from -10 mV to -40 mV, the electrostatic force increases by one order of magnitude and becomes more significant when compared with the van der Waals force. However, the trend of electrostatic force variation with ionic strength remains unchanged; (10) as pH increases, the electrostatic force increases, and ionic strength has a greater effect on the mobilization of particles; (11) changes in fluid temperature between 0°C and 50°C do not significantly affect the electrostatic force with various ionic strength and zeta potential.
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Chapter 1

Research Motivation, Objectives, and Literature Review

This dissertation encompasses an experimental investigation of the relative and interactive effects of certain physicochemical fluid characteristics on the incipient motion of granular particles. In this chapter, the research objective and its motivation are outlined in light of a review of relevant literature and of an identified knowledge gap. Previous studies on fluid-induced particle mobilization and its relevance to soil erosion within the field of geotechnical engineering are highlighted and past research that preliminarily revealed the influence of certain physicochemical fluid characteristics on particle mobilization and soil erosion are presented.

1.1 Research Motivation and Objective

The annual global soil sediment flux as a result of surface erosion was conservatively estimated to be 20 billion tons (Milliman and Syvitski 1992). In the United States alone, the Natural Resources Conservation Services (NRCS) reported a loss of 1.67 billion tons of agricultural topsoil due to water and wind erosion in 2012 (U.S. Department of Agriculture 2015). Surface erosion not only results in the loss of a precious natural resource, but it also permanently alters the ecosystem of the planet. In addition to surface erosion, another significant form of soil transport called subsurface or internal erosion can occur when fluids permeate through the pores of a soil matrix
In the case of water-retaining embankments such as dams and levees, which are essential for the safety and quality of life for people throughout the world, this form of erosion is of particular concern and many catastrophic failures have been attributed to its potentially destructive consequences including the 1972 failure of the Buffalo Creak Dam in West Virginia (Davies et al. 1972), the 1976 Teton Dam failure in Idaho (Penman 1987; Sherard 1987), the 1990 Cyanide Dam failure in North Carolina (Leonards and Deschamps 1998), and three levee breaches during Hurricane Katrina in 2005 (Seed et al. 2008a; b; Sills et al. 2008).

Studies on the subsurface erosion process frequently utilize distilled or de-ionized water as the permeating fluid, but this may not accurately represent field conditions where actual permeating fluids typically contain solutes and suspended particles of various forms across a range of concentrations. Consequently, certain physicochemical characteristics, including but not limited to, pH, ionic strength, viscosity, and density could take on a range of values in natural permeates. Briaud et al. (2008) pointed out that soil particle dislodging results from an interaction of forces, including electrical forces between particles, forces at contacts between particles, fluid pressure around particles, and shear stress around particles. The magnitudes of these forces and the nature of their interactions may depend, at least in part, on the physicochemical characteristics of the fluid. The precise particle erosion mechanisms brought on by these potentially complex and varying interactions are not fully understood. In response to this incomplete understanding, the objective of this research was to quantify the main and interactive effects of certain physicochemical fluid characteristics on the incipient motion of granular particles. The following subsections discuss these and other relevant themes in
greater detail and offer a brief overview of several important topics related to the experimental research presented later in this dissertation.

1.2 Review of Previous Research on Flow-induced Particle Incipient Motion

1.2.1 Fundamental Aspects of Particle Incipient Motion

Incipient motion of particles is also known as entrainment, dislodgement, or mobilization. It typically has three forms: sliding, rolling, and lifting. Field observations of rounded and semi-rounded particles subjected to near-threshold flow conditions showed the mode of motion is rolling (Bagnold 1973; Dancey et al. 2002; Drake et al. 1988; Francis 1973). Ling (1995) concluded that the lifting threshold is consistently higher than the rolling threshold in the hydraulically smooth and transitional flow regimes. That is, particles are mobilized first by rolling rather than by lifting. Laboratory flume tests on a single and fully exposed Teflon sphere resting on a bed of glass beads also showed rolling as the mode of entrainment (Diplas et al. 2008). In surface erosion, the incipient motion condition for granular material defines the stability of erodible beds and constitutes the central problem for sediment transport in rivers, coastal areas, and atmospheric flows (Celik et al. 2010). In subsurface erosion, knowledge of initiation of soil erosion within levees or dams is critical for the prevention of catastrophic failures of these critical infrastructures.

Particle mobilization is a fundamental aspect in the process of soil erosion and involves the balance of forces acting on individual grains within a soil matrix. Some of
the potential forces involved in the mobilization process include self-weight, buoyancy, hydrostatic fluid pressure, inter-particle contact and electrical forces, rotational resistance, and shear forces (Briaud et al. 2008; Fournier et al. 2005; Santamarina 2001). A simplified example illustrating some of these forces is shown in Figure 1-1, where those forces tending to induce particle mobilization include lift, $F_L$, and drag, $F_d$, forces, and the force tending to resist mobilization is frictional in nature, $F_f$. Bedrikovetsky et al. (2011) concluded that if the rotational moments caused by the mobilizing forces, $M_m$, exceed the resisting moments caused by the retention forces, $M_R$, a soil particle will become detached and erode. Note that Figure 1-1 presents a generalized interpretation of the orientation and magnitude of these forces and moments.

![Figure 1-1. Simplified schematic diagram outlining the interaction of forces during particle mobilization.](image)

Many researchers (Gessler 1970; Kramer 1935; Shields 1936; Simons and Richardson 1966; Tison 1953; Vanoni 1977; White 1940) have studied the initiation of particle motion in sediment beds. Shields (1936) was the first to propose a quantitative criterion based on averaged boundary shear stress. Shields’s seminal work presented the empirical relationship between the dimensionless hydraulic shear stress required for the
initiation of particle mobilization (called the Shields parameter, \( \theta \)) and the flow intensity as described by a particular form of the particle Reynolds number, \( Re_* \) (Figure 1-2). Buffington and Montgomery (1997) compiled data from eight decades of incipient motion studies and used them to calculate dimensionless critical shear stress values of the median grain size. They found that a traditional Shields diagram constructed from this data reveals systematic methodological biases of the definition of incipient motion. Despite the discrepancies between the Shields diagram and other studies (Vanoni 1964; White 1940), Shields diagram has remained the standard method for describing threshold conditions of mobile sediment for more than 70 years.

![Shields diagram](image)

**Figure 1-2.** Shields diagram, which relates the dimensionless critical hydraulic shear stress to the particle Reynolds number (Annandale 2007).

Hydraulic shear stress can be described as the tangential stress that a moving fluid exerts on a soil particle. It is caused by the viscous drag force of the fluid and has been shown to directly affect the erosion process (Gilley et al. 1994; Julian and Torres 2006; Kakuturu and Reddi 2006a; b). In their studies on clayey sand specimens, Kakuturu and
Reddi (2006a; b) found that permeating fluids with higher viscosity tend to exert higher hydraulic shear stress and cause more erosion. Other studies have focused specifically on colloidal particles and have pointed out that the critical hydraulic shear stress required to dislodge the particles from flat surfaces can vary with certain physicochemical fluid characteristics. For example, it was demonstrated that erosion potential increases with increasing fluid pH (Hubbe 1985; Sharma et al. 1992), and decreasing fluid ionic strength (McDowell-Boyer 1992) and particle size (Hubbe 1985).

In addition to the effects caused by a permeating fluid’s physicochemical characteristics, the hydraulic flow conditions are also known to affect particle mobilization. However, previous studies on the subject are in conflict. For example, in an analysis based on the work of Shields (1936), Annandale (2006, 2007) argued that the commonly held belief that soil erosion is caused by hydraulic shear stress is only valid during laminar flow; during turbulent flow, the critical hydraulic shear stress remains constant and instead varying hydrostatic lift forces caused by fluctuating fluid pressures facilitate particle detachment. In contrast to this finding, Visser (1972) concluded that lift forces contribute negligibly to particle mobilization; in both laminar and turbulent flow, shear forces govern particle mobilization. Further confounding the issue are the results of McDowell-Boyer (1992), who stated that the “mobilization process depends to a great extent on solution chemistry and that hydrodynamic forces are unimportant under steady laminar flow conditions.” Bergendahl and Grasso (2000) found the critical hydraulic shear stress to be related to the flow velocity, and recent investigations have focused on determining this critical shear stress through empirical curve fitting.
The concept of an erosion threshold has typically been applied to bench-scale studies where the correlation of hydraulic shear stresses with the resulting specimen-wide erosion rates results in the determination of a single (averaged) critical hydraulic shear stress that is applied to the gross erodibility of the entire soil specimen. For example, in their suffusion and piping erosion tests conducted using a modified triaxial apparatus, Bendahmane et al. (2008) used a turbidimeter to continuously and precisely measure erosion rate fluctuations caused by gradually increasing the hydraulic gradient. This allowed for the determination of the onset of particle movement and the subsequent quantification of the critical hydraulic shear stress.

Other methods for determining the onset of particle movement and/or critical hydraulic shear stress can also be used. For example, in the review of Shields’ (1936) work, Buffington (1999) described two methods: (1) reference techniques that involve extrapolating curves of erosion rate versus hydraulic shear stress to the point of zero erosion, which is assumed to correspond to the critical hydraulic shear stress; and (2) visual techniques that rely on the subjective determination of incipient motion based on comparisons of observed particle movements with accepted criteria, such as those proposed by Kramer (1935) and summarized by Buffington (1999). In their comparison of several popular equations relating particle detachment with hydraulic shear stress, Zhu et al. (2001) described several approaches used to quantify critical hydraulic shear stress, including two visual techniques that require determining detectable and appreciable motion. Alternatively, some researchers employed a method that involves partitioning the hydraulic shear stress into a lower and an upper range and extending the straight-line regressions for each range to their point of intersection, which corresponds to the critical
hydraulic shear stress. In light of the large degree of uncertainty related to the
determination of the critical hydraulic shear stress, Buffington and Montgomery’s (1997)
conclusion, drawn from the analysis of eight decades of research on incipient motion,
seems particularly relevant: instead of pursuing a value of critical hydraulic shear stress
that corresponds well with the work of others, researchers should place more emphasis on
defining a *defendable* critical hydraulic shear stress that is based on attention to the
particular application along with thorough considerations of the recognized limitations
associated with the particular testing methodology.

The uncertainties related to the existing studies on soil erosion suggest that the
manner in which the mobilizing forces correlate to the physicochemical characteristics of
the permeating fluid are still unclear. Apparently, the mechanisms of soil erosion
behavior may be governed, at least in part, by the potentially interacting effects imparted
by the physicochemical characteristics of the fluids and by varying hydraulic conditions.

### 1.2.2 Definitions of Critical Conditions for Particle Incipient Motion

The condition for incipient motion of particles is known as the critical or
threshold condition. Knowledge of the near-threshold flow conditions is important for a
variety of applications ranging from civil and environmental engineering to stream
ecology (Diplas et al. 2010). This threshold is governed by the relationship between the
erosive capacity of the moving fluid and the ability of the soil to resist the erosion
(Annandale 2006). In other words, the erosion threshold is the critical state for the onset
of erosion and occurs when the fluid’s erosive capacity overcomes the soil’s resistive
capacity. Review of previous research on incipient motion of particles of various scales seems to suggest a lack of consistent definition and quantification for the condition. Vanoni (1964) conducted flume experiments with two sediments, a quartz sand with geometric mean size of 0.102 mm and glass beads with geometric mean diameter of 0.037 mm. When the threshold conditions for particle mobilization were approached, intermittent sediment motion was observed. This motion occurred in bursts or sweeps produced by the turbulence in the flow near the bed. When the burst frequency fell to between 113 and one burst per second, threshold conditions for critical motion were considered to exist. In the flume tests by Shields (1936), the critical shear stress was taken as the value of the shear stress that would give zero particle mobilization and was determined by graphical extrapolation of the sediment transport rate. This approach was also adopted by other researchers such as Arulanandan et al. (1975) in rotating cylinder erosion tests. In flume tests on cohesionless, fine graded, flaked sediment, Mantz (1973) defined the incipient motion based on some minimum but consistent rate of particle mobilization, though the exact rate was not provided. In experiments on the detachment of colloidal particles (5 to 40 µm) from surfaces, Sharma et al. (1992) defined the critical condition for particle incipient motion as the point corresponding to the mobilization of 10% of the total number of particles on the bed. Lyle and Smerdon (1965) utilized the partitioning of hydraulic shear stress into a lower and an upper range, where the straight-line intersection of each range defined the critical hydraulic shear stress.
1.2.3 Intrinsic and Statistical Variability in Quantifying Particle Incipient Motion

Although experiments conducted at the scale of individual particles can be useful when studying the fundamental mechanisms of particle mobilization, they also present specific challenges. For example, because of the inherently stochastic nature of some of the major factors affecting the process of individual particle mobilization, including orientation of individual particles with respect to adjacent particles and fluctuating fluid pressures, even particles of the same size within the same particle bed can exhibit a range of behaviors (Gessler 1971; Grass 1970; Kirchner et al. 1990; Paintal 1971; Shields 1936). The field and laboratory observations reported by several researchers (Buffington et al. 1992; Kirchner et al. 1990; Lavelle and Mofjeld 1987; Wilcock 1996) also confirmed the variability of critical shear stress that could be attributed to a number of random factors. Wu and Chou (2003) listed some contributors to the stochastic nature of sediment entrainment including the temporal fluctuations of turbulent flow (Kalinske 1947), heterogeneities in grain size, shape, and density (Bridge and Bennett 1992), bed grain geometry (Naden 1987), availability of sediment (Church 1977), exposure and sheltering effects (Kirchner et al. 1990; McEwan and Heald 2001), and bed roughness (Papanicolaou et al. 2001).

Cleaver and Yates (1973) and Hubbe (1985) showed that the effect of time on the extent of detachment is due to fluctuations in local hydrodynamic forces acting upon the particles; these localized fluctuations result in progressive entrainment from surfaces exposed to constant time-averaged flow in the viscous sublayer and fluctuations of relatively long duration have significant effect. The initial bed form was also shown to
affect the critical condition for particle mobilization. The flume experiments of Vanoni (1964) showed a decrease in particle movement from the beginning to the end of the tests when flow conditions were near critical; in some cases, particle mobilization actually ceased after a period of one to two hours. This behavior was attributed to the presence of initially unstable grains near the surface of the bed left from the bed-leveling process. These grains continued to move until they found a stable position further downstream on the bed or were swept out of the flume. Because of this adjustment period, at least one hour under steady flow was allowed before observations of intensity of motion were made. This adjustment period complicates and further adds to the difficulty of quantifying the threshold condition.

This probabilistic nature complicates the task of defining an erosion threshold, which can be quantified in terms of the hydraulic shear stress acting on a particle (Buffington and Montgomery 1997). Some research has identified a range of threshold flow conditions for particle incipient motion and thus employed probabilistic models as an alternative approach to the sediment entrainment problem (Einstein 1942; Gessler 1970; Grass 1970; Wu and Chou 2003). In their studies using idealized spherical particles, Kirchner et al. (1990) found that even for particles of the same size within the same particle bed, critical hydraulic shear stress is characterized by a probability distribution rather than a single value. They attributed this particle-specific variation in critical hydraulic shear stress, at least in part, to the orientation of individual particles with respect to the surrounding particles. More specifically, critical hydraulic shear stress for individual particles is highly dependent on particle projection and exposure above mean bed height, and on particle friction angle. From this understanding, Kirchner et al.
(1990) hypothesized that conventional measurements of critical hydraulic shear stress (presumably at the bench scale for an assembly of particles) define the erosion threshold only for the most easily eroded particles. These findings suggest some of the conventional quantifications of critical hydraulic shear stress may be conservative.

Buffington and Montgomery (1997) thoroughly explained the scatter within the Shields diagram through consideration of a variety of factors, such as differences in particle bed properties (i.e., grain sorting, packing, shape, and roundness), neglect of roughness elements during analysis, method of shear stress measurement, sampling technique used to characterize grain size distributions, differences in the scale and duration of sediment transport observations, and differences in the scale at which incipient motion is defined. Real beds are heterogeneous with randomly positioned particles of varying size and as a result the relative protrusion of a particle above the bed (particle exposure) is a random variable (Lee and Balachandar 2012). This and other factors including grain shape, pocket geometry and bed geometries are known to influence the hydrodynamic forces acting on the particle, but more importantly, they strongly alter the resistive forces countering particle mobilization (Johnston et al. 1998; Kirchner et al. 1990; Schmeeckle and Nelson 2003).

The past research seems to suggest that if one requires a phenomenological description of the relationship between variables such as applied shear stress, bedload transport rate, and grain size distributions of the bedload and bed, intermediate factors such as protrusion and friction angle may perhaps be safely ignored. However, if a mechanistic understanding of the processes governing sediment transport is required, these factors demand attention by virtue of their role in controlling grain mobility.
1.3 Relevance of Particle Mobilization in the Field of Geotechnical Engineering

Particle mobilization (erosion), transport, and immobilization (clogging) have been studied in the field of geotechnical engineering to improve the understanding of subsurface erosion in water-retaining embankments such as levees and dams. Based on extensive field observations and laboratory testing spanning many decades, Terzaghi et al. (1996) discussed the various mechanisms and processes involved in a particularly destructive form of subsurface erosion called piping erosion. This type of erosion is characterized by the formation of a hollow, subsurface piping channel that begins on the downstream (dry) side of the embankment where seepage forces result in the removal of soil particles at the ground surface (Figure 1-3). This process is often accompanied by the formation of sand boils and seepage springs (Figure 1-4) and can facilitate further erosion through two predominant processes: (1) by providing an open space for the progressive removal of additional particles (Figure 1-5); and (2) by decreasing the length of the seepage path and consequently increasing the hydraulic gradient acting on the soil particles.

Figure 1-3. Subsurface erosion in water-retaining embankment.
Figure 1-4. Seepage-induced sand boils indicating the removal of soil particles and possible piping erosion.

Figure 1-5. Piping erosion progression in the direction opposite to the flow.

If erosion along the flow path progresses to the point of creating a continuous connection between the wet (upstream) and dry (downstream) sides of the embankment, the hydraulic head difference between the two sides of the structure will cause permeating fluid to rush through the open channel. Continued erosion caused by the increasing flow of permeating fluid increases the size of the piping channel. This process can eventually undermine the integrity of the embankment and ultimately lead to failure and collapse of the entire structure (Figure 1-6). Indeed, this form of erosion can be highly destructive and is known to cause catastrophic failures of earthen dams and levees (Leonards and Deschamps 1998; Seed and Duncan 1981). In fact, a survey of 11,192
dams revealed that approximately 46% of failures could be attributed to subsurface erosion (Foster et al. 2000). This high rate could be linked to the fact that the initial stages of the subsurface erosion process go largely unnoticed as they occur below the ground surface. By the time visible signs are present at the surface, failure may be imminent or at best, damages requiring mitigation and/or repair may already have occurred. Earlier detection or predictions of subsurface erosion are needed to increase the time available to intervene and to decrease the extent and cost of repairs.

![Figure 1-6](image)

Figure 1-6. Piping-erosion-induced levee failure at (a) the Kaskaskia River in southern Illinois in 1993 (Resio et al. 2009) and (b) the Upper Jones Tract levee in the California Delta in 2004 (DWR 2004).

It is important to note that, although piping erosion is considered to be a form of subsurface erosion, the interaction between the permeating fluid and the soil particles on the surface of the wall of the piping channel is similar to the hydrodynamic process of surface erosion in open channels. Therefore, certain topics related to surface erosion processes can be applied to the study of piping erosion.
1.4 Previous Research on the Effects of Physicochemical Fluid Characteristics on Particle Mobilization

In previous experimental studies on soil erosion, including flume tests, the pinhole erosion test, the hole erosion test (HET), and the slot erosion test, tap water or de-ionized water have often been used as the permeating fluid. However, when a fluid permeates through soil and interacts with the environment, its properties are altered from those of pure water (Hillel 1998). In the field, fluids permeating through earthen dams or levees may exhibit a range of chemical compositions and may also contain suspended particles of various sizes and concentrations. For example, during slurry cut-off wall installations in levees, the walls of the excavation are often stabilized by filling the open trench with bentonite slurry. This slurry could seep through the embankment itself or through an existing piping channel (Figure 1-7). Also, natural seepage in levees and earthen dams may contain dissolved solutes or fine particles that are entrained in the pore fluid during upstream interactions with the environment. Clearly, permeating fluids have the potential to exhibit varying physicochemical characteristics that differ from those of pure water. Some experiments have demonstrated that permeating fluids consisting of de-ionized water and various concentrations of sodium chloride can induce different erosion behaviors (Arulanandan et al. 1975; Reddi et al. 2000a; Yong et al. 1979) and that clogging of soil pores may depend on physicochemical characteristics of the permeating fluids (Sherard et al. 1984). Though ultimately inconclusive, results from the MS thesis of Sinco (2011) suggested that erosion behavior might be affected by interactions between the permeating fluids’ pH, ionic strength, viscosity, and suspended solids
composition (plastic and non-plastic) and concentration and that further research is needed to develop an improved understanding of the topic.

Figure 1-7. Schematic representation of the potential seepage of slurry through an existing piping channel.

Significant research has been conducted in the past to investigate the effects of physicochemical characteristics of fluids (with focus on pH and ionic strength) on the incipient motions of colloidal-size (<1 μm) particles. These studies generally concluded that higher pH and lower ionic strength resulted in easier particle mobilization. For example, Sharma et al. (1992) experimentally studied the effect of pH on the release of 10-μm glass microspheres from a glass surface for solution pH values of 5, 7, and 10. They concluded that increasing pH increases the probability that a glass particle will be detached at a given hydrodynamic force, since the glass surface becomes more negatively charged at higher pH values. They also conducted experiments in a glass cell using 10-μm glass microspheres and solutions with different concentrations of NaCl. The results showed that the effect of ionic strength is not as large as that of pH; decreasing ionic strength causes only a marginal decrease in the hydrodynamic force required for release. Hubbe (1985) experimentally studied the detachment of colloidal titanium hydrous oxide
spheres from cellulose and glass substrates exposed to turbulent shear flow. He found the shear stress required for detachment depended strongly on the size of the particles and on the pH of the solution; increasing pH corresponded to a decrease in the shear stress required for detachment. Visser (1972) stated this effect may be attributed to changes in the electrostatic forces arising from the overlap of ionic atmospheres at the charged interfaces. Kolakowski and Matijevic (1979) and Kuo and Matijevic (1980) showed that the release of hydrous oxide spheres from packed columns of glass and steel beads was a function of pH, ionic strength, and specifically adsorbed ions. Visser (1970, 1976) showed that the hydrodynamic shear stress required to detach carbon black and polystyrene spheres from cellulose was a function of pH and ionic strength. Using laboratory sand column tests, McDowell-Boyer (1992) observed that the mobilization process of 1.46-µm polystyrene latex particles in a saturated porous media depends to a great extent on solution chemistry and concluded that “under steady, laminar flow conditions, hydrodynamic drag and lift forces are likely unimportant with respect to chemical mobilization forces” for colloidal particles.

Limited research has been conducted on the effects of permeating fluid’s physicochemical characteristics on soil’s erosion behaviors at the bench scale. Using flow pump tests on a sand-kaolinite mixture and permeating fluids with ionic strength of 0, 0.001 N, 0.01 N, Reddi et al. (2000a) found higher ionic strength causes higher critical velocity (lower erosion), for both hole erosion (piping) and subsurface erosion (suffusion) tests. Sherard et al. (1972) reported a main factor in the susceptibility of the clay mass to erosion is the total content of the dissolved salt in the eroding fluid; the lower the content of dissolved salts in the water, the greater the susceptibility of clay soil to erode. The salt
content is directly related to ionic strength. Arulanandan et al. (1975) used a rotating cylinder test apparatus to measure the critical shear stress of a cohesive loam and found the salt concentration of the eroding fluid can have a significant effect on the critical shear stress for surface erosion; the erodibility of a soil decreases with the increase of NaCl concentration. It seems that for both colloidal and granular particles, increasing an eroding fluid’s ionic strength tends to decrease its erosive capacity.

Previous research preliminarily revealed the individual effects of a fluid’s viscosity, pH, and ionic strength on the mobilization of colloidal particles and the erosion of clayey soils. Whether and how these physicochemical characteristics affect the incipient motion of granular particles (individual grain diameters on the order of millimeters) is unknown. The effect of these physicochemical characteristics may also be interactive in that the magnitude of their individual effects may be dependent on the levels of the other characteristics. The relative and interactive effects of fluid physicochemical characteristics on the incipient motion of granular particles under laminar flow conditions are the focus of this research.
Chapter 2

Evolution of Research Methodology

The experimental design and methodology employed in conducting this research were developed through an evolutionary process focused on creating the ability to robustly quantify how certain fluid characteristics affect the incipient motion of a granular test particle under laminar flow conditions. In this chapter’s discussion of the research methods, four distinct phases of this evolutionary process are presented and focus is placed on how new insights and understanding gained in each phase informed the progression through subsequent phases. The fourth and final phase represents the most recent advancements and it is with this phase’s methodology that the data used to generate the results presented in Chapter 3 were gathered. For the preceding phases, preliminary results used in directing the evolutionary developments of the methodology are presented here in Chapter 2. All phases shared in common the use of an imaging system to observe and/or record the behavior of the small test particles. The main aspects that underwent progressive developments throughout the four phases were the granular test particles, the flow cell that contained the test particles during testing, and the flow system that provided and regulated the flow of the test fluids upon the test particles. Each of these aspects and their evolutionary developments will be elaborated upon in the discussion of each of the four phases.
2.1 Phase 1 – Spherical Test Particles, a Flat Flow Bed, Syringe Pumps, & a Full-factorial Designed Experiment

2.1.1 Overview of Phase 1 Experimental Equipment and Setup

An overview of the Phase 1 experimental setup is shown in Figure 2-1. The imaging system was comprised of a high-speed, high-resolution digital camera (model: ORCA®-flash2.8; manufacturer: Hamamatsu Corporation) outfitted with a thread-connected zoom lens (model: ZOOM 7000; manufacturer: Navitar) capable of capturing clear images of the test particles (Figure 2-2c). Acquiring such images required high intensity lighting. Therefore, the transparent flow cell rested atop a transmitted-light base (model: MDG 33; manufacturer: Leica Microsystems) that provided backlighting of the flow cell and test particles and created high-contrast images. The imaging system was operated using Hamamatsu Corporation’s image acquisition software called HCImage Live. The software allowed for manipulation of the frame rate, duration of acquisition, exposure settings, etc., and allowed for image sequences to be exported in a variety of formats. Image processing and analyses were carried out using the National Institutes of Health’s free, open source software called ImageJ. A system of two programmable syringe pumps (base model: NE-1000; manufacturer: New Era Pump Systems Inc.) were interconnected with a valved plumbing system and transferred fluid from a reservoir to the custom-fabricated, acrylic flow cell. Each pump was equipped with two 140 cm³ syringes, though the actual maximum volume was limited by the pumps’ physical dimensions to approximately 120 cm³ per syringe.
Figure 2-1. Overview of the Phase 1 experimental setup in (a) schematic and (b) photographic form. Note that in these images, only one of the two syringe pumps is shown.

The design and manufacturing of the flow cell considered the following requirements: (1) the top and bottom should be transparent to allow lights from both sides
to illuminate the test particles and allow for clear images of particle movements to be captured; (2) it should generate a steady and laminar flow across the entire cross section of the particle-containment area; (3) the area around the test particles should be large enough for the particles to become mobilized and enter the flow-stream without impediment from the boundaries of the particle-containment area, but small enough to generate sufficient flow velocity (and hence shear stress) to produce particle mobilization; (4) it should allow for consistent and repeatable setup of the test particles between test trials; and (5) it should allow for the removal of air during the initial saturation process.

The flow cell (Figure 2-2a, b) was constructed as two components: (1) a rectangular, open-topped box; and (2) a removable top plate. The box had inner dimensions of 2.5 cm × 7.6 cm × 21.5 cm. A trapezoidal block, constructed of the same acrylic material used to construct the flow cell, was permanently bonded to the bottom of the box and provided the smooth, flat bed for the test particles. With a rubber gasket and the top plate in place, the height of the flow area around the test particles was 0.35 cm. Rubber tubing with an inside diameter of 0.8 cm provided flow into and out of the cell through hose-barb connections at either longitudinal end. The portion of the flow cell upstream of the trapezoidal block was packed with glass spheres ranging between 0.6 and 0.9 cm (nominal) in diameter to distribute the incoming jet-like flow before it reached the test particles. The top plate included a valve to facilitate easy removal of air during the initial saturation process. Square notches were drilled around the perimeter of the bottom half of the flow cell and housed metal nuts. These metal nuts aligned with bolts passing
through holes drilled around the perimeter of the top plate and served to hold the two halves of the flow cell together.

Figure 2-2. Custom-fabricated flow cell in (a) schematic and (b) photographic form. At the start of a test, a line of test particles was placed perpendicular to the flow direction within the camera’s field of view (c).

For the Phase 1 trial tests, three nominal size categories (0.1 cm, 0.2 cm, and 0.3 cm; manufacturer: Glen Mills Inc, Clifton, New Jersey, USA) soda-lime glass beads were used as the test particles (Figure 2-2c). These particles were chosen because of their relative uniformity in terms of roundness and size distribution and the hope that this would decrease the variability of the critical flow velocity and provide greater power to
detect differences resulting specifically from changes in the test fluids’ physicochemical characteristics.

2.1.2 Phase 1 Statistical Design of Experiment and Test Fluid Compositions

A full factorial designed experiment was initially developed to form the guidelines for the creation of the test fluids and their implementation in the testing program. However, initial trial testing of the methodology using room temperature distilled water revealed issues with reliability and repeatability of the results. This obviated the ability to proceed with the initial plan for the full experimental program and instead set the stage for the progressive developments to improve the methodology. Nonetheless, this initial plan is presented here as some of its aspects were carried over into the subsequent developmental phases.

With four experimental factors (i.e. fluid physicochemical characteristics) under consideration (suspended solids concentration, pH, ionic strength, and viscosity) tested at two levels each (a low level and a high level), a total of $2^4=16$ factor-level combinations were generated for the full factorial design; that is, the experimental design called for the testing of 16 different fluids, each with a unique combination of the four physicochemical characteristics. This type of design allowed for the linear estimation of the factors’ potential effects and if the initial analysis indicated possible curvature in the response, additional data that included the relevant centerpoints could be collected to facilitate additional and more appropriate analyses. Additional experiments with more extreme high and low levels of the factors could also be included. Statistical analysis of the data
was to be performed through the fitting of a statistical model using the analysis of variance (ANOVA) technique to identify/quantify the main and interactive effects of each factor. Graphical analyses and presentation of the results could also be generated.

The four experimental factors and their high and low levels are outlined in Table 2-1. In the case of pH and viscosity, the targeted values were arbitrarily selected to cover a relatively broad range of potential values for each property. For ionic strength, the low-level target was selected based on the minimum possible value that could be achieved after adjusting the fluids to the targeted pH values through the addition of acid or base. The high-level target for ionic strength was selected to model the situation where seawater (aquion 2014) or some other similarly high-ionic-strength solution acts as the permeating fluid. For the suspended solids concentrations, the low-level target was selected to model the situation where no or very few suspended solids are present in the permeating fluid, while the high-level target was specified based on the concentration of bentonite clay often found in the slurry used during slurry cutoff wall installation.

Table 2-1. Table of Factors and their Test Levels

<table>
<thead>
<tr>
<th>Factor</th>
<th>Low Level</th>
<th>High Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Suspended Solids Concentration (%)</td>
<td>0.0</td>
<td>6.0</td>
</tr>
<tr>
<td>pH</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>Ionic Strength (mol/L)</td>
<td>0.004</td>
<td>0.7</td>
</tr>
<tr>
<td>Dynamic Viscosity (g/cm·s)</td>
<td>0.005</td>
<td>0.013</td>
</tr>
</tbody>
</table>

Table 2-2 presents a hybrid design and partial model matrix in standard (i.e. non-randomized) order, where all of the $2^4=16$ possible factor-level combinations are specified.
Distilled water was to be used as the base of each test fluid. Then, following a particular sequence (explained later), specific additives were to be incorporated to achieve the targeted levels of each of the four physicochemical characteristics.

The targeted suspended solids concentration was to be achieved by simply adding the appropriate mass of bentonite clay to the fluid. If the clay presented difficulties due to mineral dissociation and resulted in an adverse effect on the ability to achieve the targeted ionic strength and pH, monodisperse (mean particle diameter ≈ 10 µm) polystyrene microspheres could be used as an alternative source of suspended solids. These particles have been used as suspensions in particle clogging experiments in sand and geotextiles (Reddi et al. 2000b; Xiao 2001).

The pH was to be lowered to the targeted value of 4 by adding an appropriate quantity of hydrochloric acid (HCl), and raised to the targeted value of 10 by adding an appropriate quantity of sodium hydroxide (NaOH). Hydrochloric acid and NaOH are examples of a strong acid and base, respectively, which are known to dissociate completely in water and therefore yield reliable concentrations of hydrogen ions (and thus pH) at equilibrium (Benjamin 2010). Theoretically, the required amount of acid or base can be determined with the following mass-balance equation:

\[ M_1 V_1 = M_2 V_2 \]
Table 2-2. Standard Order Design Matrix for Four-factor, Two-level Experimental Design

<table>
<thead>
<tr>
<th>Fluid Number</th>
<th>Suspended Solids Concentration (%)</th>
<th>pH</th>
<th>Ionic Strength (mol/L)</th>
<th>Viscosity (g/cm·s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0</td>
<td>4</td>
<td>0.004</td>
<td>0.005</td>
</tr>
<tr>
<td>2</td>
<td>6.0</td>
<td>4</td>
<td>0.004</td>
<td>0.005</td>
</tr>
<tr>
<td>3</td>
<td>0.0</td>
<td>10</td>
<td>0.004</td>
<td>0.005</td>
</tr>
<tr>
<td>4</td>
<td>6.0</td>
<td>10</td>
<td>0.004</td>
<td>0.005</td>
</tr>
<tr>
<td>5</td>
<td>0.0</td>
<td>4</td>
<td>0.7</td>
<td>0.005</td>
</tr>
<tr>
<td>6</td>
<td>6.0</td>
<td>4</td>
<td>0.7</td>
<td>0.005</td>
</tr>
<tr>
<td>7</td>
<td>0.0</td>
<td>10</td>
<td>0.7</td>
<td>0.005</td>
</tr>
<tr>
<td>8</td>
<td>6.0</td>
<td>10</td>
<td>0.7</td>
<td>0.005</td>
</tr>
<tr>
<td>9</td>
<td>0.0</td>
<td>4</td>
<td>0.004</td>
<td>0.013</td>
</tr>
<tr>
<td>10</td>
<td>6.0</td>
<td>4</td>
<td>0.004</td>
<td>0.013</td>
</tr>
<tr>
<td>11</td>
<td>0.0</td>
<td>10</td>
<td>0.004</td>
<td>0.013</td>
</tr>
<tr>
<td>12</td>
<td>6.0</td>
<td>10</td>
<td>0.004</td>
<td>0.013</td>
</tr>
<tr>
<td>13</td>
<td>0.0</td>
<td>4</td>
<td>0.7</td>
<td>0.013</td>
</tr>
<tr>
<td>14</td>
<td>6.0</td>
<td>4</td>
<td>0.7</td>
<td>0.013</td>
</tr>
<tr>
<td>15</td>
<td>0.0</td>
<td>10</td>
<td>0.7</td>
<td>0.013</td>
</tr>
<tr>
<td>16</td>
<td>6.0</td>
<td>10</td>
<td>0.7</td>
<td>0.013</td>
</tr>
</tbody>
</table>

Where \( M_1 \) = molar concentration of strong acid or base

\( V_1 \) = volume of strong acid or base (i.e. the required volume)

\( M_2 \) = targeted molar concentration of final solution

\( V_2 \) = required volume of final solution

The molar concentration can be expressed in terms of pH by:

\[
\text{pH} = - \log_{10}[H^+] \quad (2)
\]
Where \( [H^+] = \) concentration of hydrogen ions in the solution

In the case of the NaOH, this method could be used by considering the relationship between the concentrations of hydrogen ions and hydroxide ions, as expressed by the equilibrium constant in the equilibrium equation for the dissociation of water (Benjamin 2010):

\[
K_w = [H^+][OH^-] = 10^{-14}
\]  

(3)

Where \( K_w = \) equilibrium constant for the dissociation of water

\([OH^-] = \) concentration of hydroxide ions in the solution

In practice, however, it could be more convenient to simply measure the solution’s pH while simultaneously adding small quantities of the acid or base until the desired pH is achieved.

A similar incremental approach could be used to adjust the solution to the desired ionic strength. Here, sodium chloride (NaCl) was to be used because it effectively alters the ionic strength with little or no effect on pH. The reciprocal relationship, however, does not hold; adding acids or bases to a solution necessarily alters the ionic strength through the addition of hydrogen and hydroxide ions. The magnitude of this effect and the required adjustment to reach the targeted ionic strength can be determined by the following equation (Benjamin 2010):

\[
I = \frac{1}{2} \sum_{\text{all ions}} c_i \cdot z_i^2
\]  

(4)
Where \( I \) = ionic strength of the solution

\( c_i \) = concentration of the \( i^{th} \) ionic species

\( z_i \) = charge on the \( i^{th} \) ionic species

The targeted viscosities were to be achieved by controlling the temperature of the fluid using a temperature control bath.

Based on the preceding discussion, it can be seen that achieving all of the factor-level combinations would require that the modifications to the distilled water be performed in a specific order. First, the suspended particles (either bentonite or polystyrene microspheres) must be added to the distilled water. The resulting effect on pH could be measured and then adjusted up or down to the targeted value accordingly. Then, the ionic strength of the mixture could be measured and increased to the targeted value accordingly (the initial ionic strength after the addition of suspended solids and the acid or base would define the targeted low level for ionic strength). Finally, this mixture’s viscosity could be measured and adjusted accordingly by either increasing or decreasing the fluid temperature. The specific composition of each of the 16 test fluids is described in Table 2-3, where the modifications to the distilled water are listed in the order in which they were to be performed. After preparing initial trial mixtures of each fluid, the modifications described in the table could be quantified and standardized to facilitate future preparations of the fluids.

Standard techniques were to be used to verify the actual levels of each permeating fluid’s physicochemical characteristics. Ionic strength and pH were to be assessed using the Fisher Scientific accumet ® model 25 pH/ion meter available at Penn State’s Civil
Infrastructure Testing and Evaluation Laboratory (CITEL). External consultants at Penn State’s Materials Characterization Laboratory could determine fluid viscosities. Fluid densities were to be measured with the Fisher Scientific Precision Specific Gravity Hydrometers, which are accurate to within 0.001 specific gravity units. The suspended solids concentrations were to be assessed using an optical turbidimeter installed just downstream of the flow cell.

The sample size (i.e. the number of unique test particles that must be assessed), \( n \), for each permeating fluid was to be determined by considering two aspects of the response variable (e.g. critical fluid velocity): (1) the tolerable error (i.e. the width of the confidence interval about the sample mean); and (2) the level of confidence in the calculated interval. The equation for calculating the required sample size is thus:

\[
\begin{align*}
n &= \frac{(z_{\alpha/2})^2 \sigma^2}{E^2} \\
\end{align*}
\]  

(5)

Where 

\[
\begin{align*}
\alpha &= \text{standard } z\text{-value corresponding to the desired confidence level} \\
\sigma &= \text{population standard deviation of critical velocity} \\
E &= \text{half-width of the confidence interval around average critical velocity}
\end{align*}
\]
Table 2-3. Descriptions of Permeating Fluid Compositions

<table>
<thead>
<tr>
<th>Fluid Number</th>
<th>Permeating Fluid Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Distilled water + HCl + Temperature Increase</td>
</tr>
<tr>
<td>2</td>
<td>Distilled water + Suspended Solids + HCl + Temperature Increase</td>
</tr>
<tr>
<td>3</td>
<td>Distilled water + NaOH + Temperature Increase</td>
</tr>
<tr>
<td>4</td>
<td>Distilled water + Suspended Solids + NaOH + Temperature Increase</td>
</tr>
<tr>
<td>5</td>
<td>Distilled water + HCl + NaCl + Temperature Increase</td>
</tr>
<tr>
<td>6</td>
<td>Distilled water + Suspended Solids + HCl + NaCl + Temperature Increase</td>
</tr>
<tr>
<td>7</td>
<td>Distilled water + NaOH + NaCl + Temperature Increase</td>
</tr>
<tr>
<td>8</td>
<td>Distilled water + Suspended Solids + NaOH + NaCl + Temperature Increase</td>
</tr>
<tr>
<td>9</td>
<td>Distilled water + HCl + Temperature Decrease</td>
</tr>
<tr>
<td>10</td>
<td>Distilled water + Suspended Solids + HCl + Temperature Decrease</td>
</tr>
<tr>
<td>11</td>
<td>Distilled water + NaOH + Temperature Decrease</td>
</tr>
<tr>
<td>12</td>
<td>Distilled water + Suspended Solids + NaOH + Temperature Decrease</td>
</tr>
<tr>
<td>13</td>
<td>Distilled water + HCl + NaCl + Temperature Decrease</td>
</tr>
<tr>
<td>14</td>
<td>Distilled water + Suspended Solids + HCl + NaCl + Temperature Decrease</td>
</tr>
<tr>
<td>15</td>
<td>Distilled water + NaOH + NaCl + Temperature Decrease</td>
</tr>
<tr>
<td>16</td>
<td>Distilled water + Suspended Solids + NaOH + NaCl + Temperature Decrease</td>
</tr>
</tbody>
</table>

Because the population standard deviation is not known, it must be estimated using the sample standard deviation that can be determined through trial testing. These initial trials could also provide a frame of reference for specifying the desired width of the confidence interval (e.g. the trial sample mean ± 10%). A confidence level of 95% was to be used.
2.1.3 Phase 1 Experimental Procedure and Method of Analysis

A typical experimental trial was prepared by manually placing the test particles atop the trapezoidal block within the flow cell in an orientation similar to that shown in Figure 2-2c. Depending on their size, as many as 15 test particles (with an average inter-particle spacing of approximately 1 mm) could fit within the camera’s field-of-view. This specific test-particle orientation was selected over a traditional particle bed for two main reasons: (1) to reduce or perhaps eliminate the variability of the initiation of motion caused by non-uniform and random inter-particle contact orientations and thus allow for the effect of the fluid characteristics to be more directly investigated; and (2) the single line of test particles oriented perpendicular to the direction of flow could help ensure that the same average flow velocity arrived at each particle without hindrance from other upstream particles or obstructions. Another anticipated benefit of this approach was that by testing many particles in a single experimental run, a statistically significant amount of data (where the critical velocity for each particle constituted an individual data point) could more readily be acquired for each of the test fluids.

After attaching the top plate and connecting the plumbing, and with the aid of the air-release valve, the flow cell was carefully filled with fluid in a manner that disturbed the row of test particles as little as possible. Because of the exploratory and validative nature of the Phase 1 tests, room temperature distilled water was ultimately used as the only test fluid in an effort to maintain consistency and minimize the potential impact on the critical velocity caused by variations in the fluid’s physicochemical characteristics. Each trial test was initiated by activating the syringe pumps, which were programmed to
operate in the so-called “dual pumps” mode where one pump acted as a “master” and the other as a “slave” that simultaneously copied the behavior of the master (Figure 2-3). In this tandem mode, the total flow rate was equal to the sum provided by the two individual pumps, and each pump program was limited to a maximum volume of approximately 480 cm$^3$ (120 cm$^3$ per syringe times four syringes) available from a single “infusion” of the syringes, i.e. refilling of the syringes was not possible as the flow rate would drop to zero during the refilling. An additional limitation on the pumps was their ability to overcome the backpressure caused by hydraulic friction throughout the flow system. With some degree of variation depending on the exact configuration of the system, this translated to a maximum flow rate of approximately 30 cm$^3$/s. In all of the Phase 1 tests, the pumps were programmed to provide a flow rate that began at zero and increased at a known, constant rate. Included within each pump program was an appropriately-timed electrical trigger signal (sent from the pump via a wired TTL-I/O connection) that was received by the camera and initiated the acquisition of images. The test progressed as the flow rate steadily increased and images were acquired at a constant frame rate. Each test particle was expected to become mobilized when the flow rate (and thus flow velocity and shear stress) increased to the point where the resulting shear stress overcame the particle’s inherent ability to resist mobilization (i.e. when the flow rate/flow velocity/shearing force/shear stress reached the particle’s critical level). Each trial was concluded after all test particles became mobilized or the pump program ended, whichever came first.
The actual size of each individual test particle was estimated using image analysis techniques. As the particles became mobilized during a test and rolled downstream along the flat bed, many different angles of their apparent cross-sectional areas were presented to the camera and recorded in the image sequences. Spatial scaling of the images allowed for these areas to be quantified and used to estimate the particle diameters. With this knowledge, the fluid velocity acting at the center of each test particle was determined based on the well-known pressure-driven Poiseuille flow between parallel boundaries. For a given point far from the lateral flow boundaries (e.g. in the center of the flow cell, perpendicular to the flow direction), the vertical flow-velocity profile perpendicular to the direction of flow resembles that shown in Figure 2-4, where the maximum flow velocity, $u_{max}$, occurs at the midpoint between the lower particle-supporting block and the upper top plate and follows a parabolically decreasing profile to the point of zero velocity at the interface with each boundary. In this research, it was assumed all of the test fluids followed this characteristic parabolic velocity profile.
Figure 2-4. Parabolic flow-velocity profile of pressure-driven Poiseuille flow between the flow cell’s (parallel) particle-supporting block and top plate.

Equation (6) describes the flow velocity, \( u \), along the parabolic velocity profile at any distance, \( y \), above or below the centerline of maximum flow velocity:

\[
  u = u_{\text{max}} \left( 1 - \left( \frac{2y}{h} \right)^2 \right) \tag{6}
\]

Where 
- \( u = \) flow velocity at a location \( y \) above or below centerline of maximum flow velocity
- \( u_{\text{max}} = \) maximum flow velocity, which occurs at the centerline of the flow profile
- \( y = \) distance from centerline of maximum flow velocity to location of interest
- \( h = \) distance between the upper and lower boundaries

Equation (6) can be rewritten in terms of the average flow velocity, \( u_{\text{ave}} \), (as determined by the ratio of the pump’s flow rate to the known cross-sectional area of the flow cell’s particle-containment region) rather than the maximum flow velocity, \( u_{\text{max}} \). This is done by integrating with respect to \( y \), evaluating the result within the upper and
lower boundaries of the flow area, and setting the outcome equal to the product of the average flow velocity and the total distance between the upper and lower boundaries:

\[
\int u \cdot dy = \int_{-h/2}^{+h/2} u_{max} \left( 1 - \left( \frac{2y}{h} \right)^2 \right) \cdot dy = u_{ave} \cdot h
\]

(7)

\[u_{max} = \frac{3}{2} u_{ave}\]

(8)

\[u = \frac{3}{2} u_{ave} \left( 1 - \left( \frac{2y}{h} \right)^2 \right)\]

(9)

This method relied on determining the flow rate at the moment each particle became mobilized. Because the pump program (which described the flow rate as a function of time) and the image sequence were synchronized, the elapsed time corresponding to the mobilization of a particle was the only information needed to determine the corresponding critical flow rate. This elapsed time was determined for each particle by manually reviewing the time-stamped image sequences recorded during each test.

Two additional quantitative assessments or verifications of the flow conditions were made: (1) the flow Reynolds number, \(R_e\), and (2) the required entry length for the full development of the parabolic velocity profile between the parallel plates of the test particle containment region, \(l_e\). The general equation for the boundary Reynolds number (Munson et al. 2006) was used to calculate the flow Reynolds number with the height of the flow area, \(h\), taken as the characteristic dimension and the critical velocity for each particle used as the velocity term:
$R_e = \frac{u \cdot h \cdot \rho_f}{\mu}$

Where $\rho_f = \text{fluid density}$

$\mu = \text{dynamic viscosity}$

Equation (11) was used to calculate the required entry length to achieve fully developed flow (Munson et al. 2006):

$l_e = 0.06 \cdot h \cdot R_e$

2.1.4 Phase 1 Results and Identified Need for Improvements

Following the experimental procedure and method of analysis described in the previous section, three initial trial tests were conducted. Each trial utilized the same pump program (labeled “pp2” for pump program 2), which was designed to generate a flow velocity within the flow cell’s test particle containment region that increased linearly from 0 cm/s to 11.7 cm/s at a rate of $du = 0.81$ cm/s/s, and test particles with nominal diameters of 0.1 cm. The outcome of these trials is presented graphically in Figure 2-5 where the solid line corresponds to the changing flow velocity and the markers represent individual particle mobilizations. Each of the three marker styles corresponds to one of the three trials. Each trial showed a relatively large spread of critical velocity; in the most extreme case (Trial 38), the maximum critical velocity was nearly three times as large as the minimum critical velocity. Also, not shown on the graph is the fact that some of the
test particles never became mobilized even after the pump’s maximum output of 11.7 cm/s was reached.

Figure 2-5. Critical flow velocity for three repeat trials using particles with nominal diameter of $\phi = 0.1$ cm and pump program 2 with flow velocity increasing linearly at a rate of $du = 0.81$ cm/s/s.

It was hypothesized that perhaps the particle diameters were not as uniform as originally thought and that subtle differences in particle sizes might explain some of the variation. However, for this to be true, one would expect to see a direct relationship between particle diameter and critical velocity and Figure 2-6 shows that no such trend exists. In this graph, each marker represents an individual particle’s critical velocity where again each marker style corresponds to a different trial.
Figure 2-6. Random variation of critical flow velocity with particle diameter.

Apparently, some other factors were introducing variation into the system’s response so two additional trials were conducted to investigate further. In these trials, the same nominal particle size of $\bar{\Omega} = 0.1 \text{ cm}$ was used but a new pump program (pump program 4 or pp4) was developed with a decrease in the flow velocity’s rate of change from $du = 0.81 \text{ cm/s/s}$ to $du = 0.45 \text{ cm/s/s}$ (the maximum flow rate was increased slightly from 11.7 cm/s to 12.4 cm/s). The idea was that perhaps the original rate of change was great enough to introduce excessively high fluid accelerations that resulted in unwanted turbulent fluctuations and velocity spikes within the vicinity of the test particles. This could cause some of the particles to mobilize prematurely and result in an artificially large spread of the computed critical velocities. The outcome of these two trials is shown in Figure 2-7 where again a relatively large range of critical velocities was observed.
Figure 2-7. Critical flow velocity for two repeat trials using particles with nominal diameter of $\Phi = 0.1$ cm and pump program 4 with flow velocity increasing linearly at a rate of $du = 0.45$ cm/s/s.

As with the first three trials, some of the test particles never mobilized before the end of the pump program was reached (this is not shown in the graph), and the spread of critical velocity of those particles that did mobilize was still relatively large. However, in this case the spread from the highest to lowest critical velocity showed a factor of only about two (Trial 41) rather than the factor of three observed previously; the slower rate of change may have produced more uniform flow velocity around the particles. In looking at the relationship between critical velocity and particle size, the expected direct relationship was once again not seen (Figure 2-8).
Figure 2-8. Random variation of critical flow velocity with particle diameter.

To investigate any possible effects related to the size of the particles used in the initial trials, several more tests were performed using two additional nominal particle sizes of Ø = 0.2 cm and Ø = 0.3 cm. In these trials, pump program 4 with $du = 0.45$ cm/s/s was used. Following the style of the preceding trials, the results are presented graphically for a single trial of the Ø = 0.2 cm particles (Figure 2-9) and for two trials of the Ø = 0.3 cm particles (Figure 2-10).

Figure 2-9. Critical flow velocity for a single trial using particles with nominal diameter of Ø = 0.2 cm and pump program 4 with flow velocity increasing linearly at a rate of $du = 0.45$ cm/s/s.
Critical flow velocity for two repeat trials using particles with nominal diameter of $\bar{\phi} = 0.3$ cm and pump program 4 with flow velocity increasing linearly at a rate of $du = 0.45$ cm/s/s. The absence of any clear evidence of a direct relationship between particle size and critical flow rate for both the $\bar{\phi} = 0.2$ cm particles (Figure 2-11) and the $\bar{\phi} = 0.3$ cm particles (Figure 2-12) remained.
One final and puzzling aspect of these results that further suggested the need for improvements to the methodology was in the comparison of the average critical velocity between all trials. It could be argued that within each of the three size categories, the subtle differences in particle size (no more than 22% for any of the trials when computed as the ratio of the range of diameters within a trial to the average diameter within the same trial) were too small to have had any significant (or measurable) effect on the critical velocity and thus the expected direct relationship between particle size and critical velocity was not observed. However, such an argument would be more difficult to make when comparing the three size categories where the average size of the particle almost tripled from the smallest category to the largest. This assessment is presented graphically in Figure 2-13 and summarized in Table 2-4 where the range of each trial’s particle diameters is described in terms of two standard deviations, $s$, and the average critical velocities are reported along with their 95% confidence intervals, standard deviations, and coefficients of variation (COV). In general, the spread of the critical velocity data
within each trial was surprisingly large considering how little variation there was in the particle sizes. Interestingly, the data also suggested an inverse rather than direct relationship between particle diameter and critical velocity. However, the limited quantity of data used in these assessments (in terms of number of trials and number of observations per trial) precluded any strong inferences on such relationships and it was ultimately concluded that some lurking variable(s) and/or statistical artifacts were interfering with the reliable quantification of the critical velocity.

Table 2-4. Summary of Phase 1 Trial Test Results

<table>
<thead>
<tr>
<th>Trial</th>
<th>Pump Program &amp; Velocity Rate of Change (cm/s/s)</th>
<th>Number of Observations</th>
<th>Average Particle Diameter ± 2s (cm)</th>
<th>Critical Velocity</th>
<th>95% CI for Average (cm/s)</th>
<th>Standard Deviation (cm/s)</th>
<th>COV</th>
<th>Average Critical Flow Reynold's Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>37</td>
<td>pp2, 0.81</td>
<td>14</td>
<td>0.118 ± 0.017</td>
<td></td>
<td>7.7 ± 1.1</td>
<td>1.94</td>
<td>0.25</td>
<td>270</td>
</tr>
<tr>
<td>38</td>
<td>pp2, 0.81</td>
<td>9</td>
<td>0.119 ± 0.018</td>
<td></td>
<td>7.9 ± 2.2</td>
<td>2.83</td>
<td>0.36</td>
<td>280</td>
</tr>
<tr>
<td>39</td>
<td>pp2, 0.81</td>
<td>5</td>
<td>0.122 ± 0.019</td>
<td></td>
<td>8.6 ± 2.4</td>
<td>1.95</td>
<td>0.23</td>
<td>310</td>
</tr>
<tr>
<td>40</td>
<td>pp4, 0.45</td>
<td>8</td>
<td>0.132 ± 0.015</td>
<td></td>
<td>9.7 ± 1.2</td>
<td>1.46</td>
<td>0.15</td>
<td>350</td>
</tr>
<tr>
<td>41</td>
<td>pp4, 0.45</td>
<td>14</td>
<td>0.123 ± 0.02</td>
<td></td>
<td>9.6 ± 0.9</td>
<td>1.61</td>
<td>0.17</td>
<td>350</td>
</tr>
<tr>
<td>42</td>
<td>pp4, 0.45</td>
<td>11</td>
<td>0.205 ± 0.026</td>
<td></td>
<td>7.9 ± 1.8</td>
<td>2.71</td>
<td>0.34</td>
<td>280</td>
</tr>
<tr>
<td>43</td>
<td>pp4, 0.45</td>
<td>9</td>
<td>0.310 ± 0.003</td>
<td></td>
<td>5.9 ± 2.0</td>
<td>2.61</td>
<td>0.44</td>
<td>210</td>
</tr>
<tr>
<td>44</td>
<td>pp4, 0.45</td>
<td>9</td>
<td>0.311 ± 0.003</td>
<td></td>
<td>5.7 ± 1.5</td>
<td>1.92</td>
<td>0.34</td>
<td>200</td>
</tr>
</tbody>
</table>
To further investigate possible sources of the observed variability and identify these lurking variables or statistical artifacts, additional thought was given to the state of flow around the test particles. The theory developed for determining the critical velocity (see Chapter 1 and Section 2.1.5 of this dissertation) and for using this as the indicator of a fluid’s relative erosive capacity was based on the assumption that uniform, smooth laminar flow acted upon the test particles within the test particle containment region of the flow cell and brought about their mobilization. However, prior to conducting the Phase 1 tests, this condition was not verified and instead it was assumed that the combination of small test particles (and thus low critical velocities), small height of the flow area, and usage of flow distribution beads upstream of the test particle containment region would be sufficient to ensure the desired type of flow reached the test particles at incipient motion. While the use of Equation (10) and average critical velocities for each trial suggested that flow conditions were well within the laminar regime (based on a value of ~1,400 as the “point” of transition between laminar and turbulent flow; see
Table 2-4 for each trial’s average Reynolds number), such an assessment really only provided a rough estimate of the overall flow conditions as it was ultimately based on the gross flow rate of the entire fluid system. As such, it was suspected it may have been incapable of fully describing the flow behavior at the scale of the individual test particles. Also, from Equation (11) and a flow Reynolds number of 45, which corresponded to the pump’s maximum output of 12.4 cm/s, the maximum entrance length required for full flow development was roughly 0.7 cm; much less than the roughly 9-cm distance between the entrance to the test particle containment region and the line of test particles. However, appropriate use of this equation required a uniform flow velocity at the entrance to the test particle containment region and the partial blockage caused by the presence of the flow distribution beads likely violated this assumption. Thus, the full extent to which this flow disturbance remained in effect within the test particle containment region was not known and may have been a contributing factor to the wide spread of the trail test critical velocities. To investigate this in greater detail and assess the uniformity of flow throughout the entire test particle containment region, a series of flow tracer dye tests were performed to visualize the flow behavior. A concentrated food coloring was dissolved in water and drawn into the pump’s syringes before being driven through the flow cell’s upstream flow distribution beads and into the test particle containment region. Tests were conducted at flow velocities of 2.0 cm/s and 9.5 cm/s and in both cases it was observed that the curved surfaces of the flow distribution beads did indeed affect the flow entering the test particle containment region (see Figure 2-14 for a sequence of photos from the 2.0 cm/s test and Figure 2-15 for the 9.5 cm/s test).
Figure 2-14. Sequence of photos from flow tracer dye test at flow velocity of 2.0 cm/s
Figure 2-15. Sequence of photos from flow tracer dye test at flow velocity of 9.5 cm/s
From the insights gained through the dye tracer tests, it was hypothesized that modifying the flow cell to include a flow-straightening device might lead to more uniform flow conditions around the test particles. To quickly and easily test this hypothesis, a simple assembly of plastic straws of approximately 0.32 cm diameter and 2 cm length was constructed and installed just downstream of the flow distribution beads and additional dye tests were performed to highlight any changes in the flow behavior. A series of representative photos for tests conducted with flow velocities of 1.3 cm/s and 7.0 cm/s are shown in Figures 2-16 and 2-17, respectively.

Use of the flow straightener appeared to diminish the lateral (perpendicular to average flow direction) components of the flow velocity and resulted in a more uniform and downstream-oriented average flow in the vicinity of the test particles. Along with the other insights gained throughout the Phase 1 testing, this information led to a fourfold plan for improving the testing methodology:

1. The test particles would be replaced with specialized, highly spherical test particles to reduce variability caused by non-uniformity of particle shape and size;

2. The flat particle bed would be replaced with a specially-designed “pocket” to support a single test particle and minimize variation in the test particle’s resistance to mobilization caused by changes at the particle—bed interface;

3. A permanent flow straightener would be incorporated between the flow distribution beads and the test particle containment region to promote straight, uniform, laminar flow acting upon the test particle; and

4. Improved pump programs would be developed to better control flow behavior.
Details of these modifications are provided in the following section along with additional preliminary results and analyses.

Figure 2-16. Sequence of photos from flow tracer dye test with flow straightener and flow velocity of 1.3 cm/s.
Figure 2-17. Sequence of photos from flow tracer dye test with flow straightener and flow velocity of 7.0 cm/s.
2.2  Phase 2 – Polished Precision Spheres, Particle Support Domes, Flow Straightener, & Advanced Pump Programs

2.2.1  Highly Spherical Polished Test Particles

The new test particles that replaced those used in the Phase 1 testing were polished soda-lime glass spheres specially designed and manufactured for applications requiring maximum uniformity between particles and minimum variation in sphericity (model: P-SLGS-2.5 0.69mm, manufacturer: Cospheric LLC, Santa Barbara, CA). The particles had densities of 2.5 g/cm$^3$ ± 0.1 g/cm$^3$ and diameters of 0.069 ± 0.002 cm. The high uniformity of these particles was expected to help reduce the variance introduced when different experimental runs were performed with different test particles or with the same test particle placed in a different orientation with respect to the particle bed and the approaching flow. This was important because it was expected (and later confirmed) that the relatively small size of the particles would invariably lead to some of them becoming lost throughout the progression of the experimental program and thus necessitate the availability of suitable replacement particles to complete the remainder of the experiments.

2.2.2  Flow Cell Insert with Flow Straightener and Test Particle Support Domes

The modifications made to the flow cell were focused on minimizing the potential impact of suspected lurking variables that could interfere with the task of robustly quantifying a fluid’s relative erosive capacity based on the concept of critical flow
velocity. Going back to Figure 1-1 where the mobilization of an individual particle was discussed in terms of a balance of mobilizing and resisting forces, it can be seen that if one wishes to study how certain factors affect critical velocity (and hence relative erosive capacity of the fluid), the resisting forces must be well controlled. Otherwise, possible effects on critical velocity resulting from changes to the mobilizing forces caused by intentionally manipulating the fluid characteristics may become inseparable from uncontrolled changes to the particle’s ability to resist mobilization. With this in mind and borrowing inspiration from the work of Celik et al. (2010, 2013), an insert was designed, professionally fabricated and incorporated into the flow cell by bonding it atop the existing trapezoidal block (Figure 2-18a). The insert added two main features to the setup: (1) a row of 29 flow straightener tubes spanning the entire width of the flow cell with a streamwise length of 2.3 cm and individual diameters of 0.17 cm (Figure 2-18b, c); and (2) a particle-supporting “pocket” in the vicinity of the location where the row of test particles had previously been placed (i.e. approximately 9 cm downstream from the outlet of the flow straightener along the centerline of the flow direction; Figure 2-19c). This particle-supporting pocket was intended to provide a consistent and repeatable initial resting position for the test particles and comprised three domes protruding from the originally flat, smooth surface of the flow bed. The specific size, protrusion from the bed surface, and orientation of the domes were intended to provide three consistent points of support for the test particle by elevating it just above the surface of the underlying flat plane (Figure 2-19b). Attachment of the flow cell’s transparent lid created an enclosed flow area surrounding the test particle that measured approximately 7.62 cm in width by 0.26 cm in height. Figure 2-18d shows a photograph of the entire flow cell.
Figure 2-18. Flow cell and insert showing (a) insert’s orientation with respect to the original flow cell, (b, c) downstream and upstream views of flow straightener, respectively, and (d) photograph of entire assembly.
2.2.3 Modified Pump Programs

In attempting to overcome the suspicion that even the lowest flow velocity rate of change used in the Phase 1 trials ($du = 0.45 \text{ cm/s/s}$) may still have been too high to produce the desired steady, uniform, laminar flow around the test particles at incipient motion, three additional “styles” of pump program were investigated. In the first style, an initial linear rate of velocity increase was followed by a characteristic “stepped” period where the flow velocity was incremented by $\Delta u = 0.02 \text{ cm/s}$, maintained for one second, and then repeatedly incremented again in the same fashion until the end of the pump
program (i.e. the syringes were depleted or the maximum backpressure was reached). The intention was to use the linear period to quickly (in terms of considering the potential for changes in temperature and thus viscosity of the planned non-ambient-temperature test fluids) and conservatively (in terms of consumption of the finite volume of fluid within the syringes) bring the flow velocity up to some sub-critical level and then “gently” reach the critical velocity during one of the sustained (and presumably steady) flow velocity steps. This method ultimately suffered from several limitations that will be discussed in an upcoming section. The second style was a “proof of concept” where, similar to the Phase 1 dual-pumps style of pump program, a purely linear change in flow rate was used but in this case the rate of change was further decreased by almost an order of magnitude. This test demonstrated the ability of a very low linear rate of change to induce particle mobilizations at a relatively repeatable critical velocity, but unfortunately also suffered from its own limitation and therefore was not adopted for further trials. Instead, it inspired the final style of pump program that took advantage of the pumps’ ability to work together in their so-called “reciprocating” mode: while one pump “infused” the contents of its syringes through the flow cell, the other pump refilled its syringes by “withdrawing” fluid from the reservoir. After each pump finished its infusion or withdrawal, the pumping directions reversed and thus a back-and-forth reciprocating cycle characterized this style of pump program. Figure 2-20 shows a photograph of the pumps in operation along with a schematic illustration of their reciprocating behavior. Like the stepped pump programs, this style suffered from disqualifying limitations (discussed later) and ushered in the abandonment of the syringe pumps altogether.
2.2.4 Phase 2 Experimental Procedure and Method of Analysis

The general test procedure and method of analysis remained relatively unchanged from that of the Phase 1 tests. The major difference was that when preparing a trial, rather than forming a row of test particles resting on the flat flow bed, a single test particle was carefully placed atop the support domes. As with Phase 1, all of the Phase 2 trials were ultimately conducted using only room temperature distilled water to once again minimize any potential impacts on incipient motion caused by alterations to the fluid characteristics and thus facilitate validation of the methodological improvements.
2.2.5 Phase 2 Results and Identified Need for Improvements

The results for the Phase 2 trials are presented graphically in a fashion similar to that of the Phase 1 testing where each solid line represents the flow velocity provided by a given pump program and the markers overlaying these solid lines correspond to particle mobilizations observed during the overlaid pump program. Because of the new test setup with only a single test particle atop the support domes in each experimental run, multiple markers on the same line represent repeated trials of the same pump program and test conditions. Figure 2-21 summarizes the results of the testing performed using the stepped pump programs. Two distinct rates for the linear portion of the programs were used: $du = 2.19$ cm/s/s and $du = 0.61$ cm/s/s. The larger of the two rates was implemented first and after observing the test particle mobilize during the linear portion of the pump program at what seemed to be an exceptionally low critical velocity of approximately 3.0 cm/s, it was once again suspected that the higher rate of change may have induced turbulent fluid accelerations and led to premature mobilization. Therefore, focus for the stepped pump programs was shifted to and henceforth remained exclusively on using the lower rate of change. Several pump programs of this form were produced with each following the same initial linear rate of increase of $du = 0.61$ cm/s/s and the differentiating factor being the flow velocity at the point of transition between the linear and the stepped periods. A summary of these results is provided in Table 2-5 along with an additional graphical view in Figure 2-22.
Figure 2-21. Graphical summary of results from Phase 2 stepped pump programs where the 0.02 cm/s steps of 1 s duration were preceded by linear rates of either $du = 2.19$ cm/s/s or $du = 0.61$ cm/s/s.

Figure 2-22. Graph of region within dashed box in Figure 2-21 highlighting the stepped portions of pump programs and the transitions between linear and stepped velocity increases.
Table 2-5. Summary of Results from Phase 2 Stepped Pump Programs with $\Delta u = 0.02$ cm/s

<table>
<thead>
<tr>
<th>Trial</th>
<th>Pump Program</th>
<th>Critical Velocity (cm/s)</th>
<th>Linear or Stepped Mobilization</th>
</tr>
</thead>
<tbody>
<tr>
<td>54</td>
<td>pp11</td>
<td>2.95</td>
<td>Linear</td>
</tr>
<tr>
<td>55</td>
<td>pp12</td>
<td>5.43</td>
<td>Linear</td>
</tr>
<tr>
<td>56</td>
<td>pp13</td>
<td>5.13</td>
<td>Linear</td>
</tr>
<tr>
<td>57</td>
<td>pp14</td>
<td>4.99</td>
<td>Stepped</td>
</tr>
<tr>
<td>58</td>
<td>pp14</td>
<td>4.92</td>
<td>Stepped</td>
</tr>
<tr>
<td>59</td>
<td>pp14</td>
<td>4.92</td>
<td>Stepped</td>
</tr>
<tr>
<td>60</td>
<td>pp16</td>
<td>&gt;5.51*</td>
<td>Stepped</td>
</tr>
<tr>
<td>61</td>
<td>pp16</td>
<td>&gt;5.51*</td>
<td>Stepped</td>
</tr>
<tr>
<td>62</td>
<td>pp17</td>
<td>5.12</td>
<td>Stepped</td>
</tr>
<tr>
<td>63</td>
<td>pp17</td>
<td>5.37</td>
<td>Stepped</td>
</tr>
<tr>
<td>64</td>
<td>pp17</td>
<td>4.71</td>
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<td>Linear</td>
</tr>
<tr>
<td>69</td>
<td>pp17</td>
<td>5.45</td>
<td>Stepped</td>
</tr>
<tr>
<td>70</td>
<td>pp17</td>
<td>4.42</td>
<td>Linear</td>
</tr>
</tbody>
</table>

95% CI for average critical velocity = 4.89 ± 0.34 cm/s  
Standard deviation of critical velocity = 0.65 cm/s  
COV = 0.13

* Particle never mobilized.

At first glance and when compared to the Phase 1 results, these results seemed promising in that they showed a lower relative spread (the coefficient of variation here was 0.13 compared to the range of between 0.15 and 0.44 in the Phase 1 trials, see Table 2-4) and the margin of error was almost three times smaller than the smallest margin of error from the Phase 1 tests. However, a closer look at the data cast doubt on their strength and on the ability of the methodology to achieve the research objectives. For
example, despite the apparent numerical consistency of the data, careful consideration of how they were obtained suggested otherwise. Although pump program 17 (pp17) produced particle mobilizations during its period of stepped velocity increases for four repeat trials (see Figure 2-23), it also produced particle mobilizations during the preceding linear portion in several others. Likewise, pump program 14 exhibited similar inconsistency. And in other trials, mobilizations were not achieved until much higher flow velocities and while still within the linear period (pp12) or not at all (pp16).

![Graph of region within dashed box in Figure 2-22.](image)

Although not able to fully explain these inconsistencies, it was hypothesized that the precise manner in which the pumps introduced the flow onto the test particles was still too rough and inconsistent between trials and thus the “proof-of-concept” pump program was designed and implemented to test this idea. Recall that the flow velocity increased linearly (starting from zero) for the entire duration of this pump program and at a rate approximately one order of magnitude lower than in the Phase 1 trials. As such, the length of time required to reach the critical velocity was increased significantly and it was
acknowledged that use of this pump program would complicate the task of maintaining consistent fluid temperatures (and thus viscosities) in the planned non-ambient-temperature test fluids. Nevertheless, trials with this pump program provided further insight into the relationship between the velocity’s rate of change and the repeatability of quantifying the critical velocity. From only three repeat trials (i.e. a very low number of observations), the 95% confidence interval for the average critical velocity was $4.73 \pm 0.38$ cm/s with a standard deviation of 0.15 cm/s and coefficient of variation of 0.03. These results are shown graphically in Figure 2-24 where the curves for pp2 and pp4 from Phase 1 are also shown for comparison.

![Figure 2-24. Graphical depiction of the “proof-of-concept” pump program (pp26) shown alongside Phase 1 programs to highlight its low rate of change of flow velocity.](image)

With this verification of the need for very low linear rates of change of velocity, the new challenge was to devise a way to achieve this while also ensuring the fluid’s residence time within the syringes wasn’t so long that excessive changes in temperature could occur. The proposed solution was to use the pumps’ reciprocating mode of operation to reach some near-but-sub-critical flow velocity and then quickly refill the
syringes with fresh fluid from the temperature-controlled reservoir to induce mobilization at the targeted temperature and viscosity. Many attempts at this were undertaken with different rates of change, sophisticated methods of creating a smooth transition between the infusion by one pump and then the other, and even several configurations of the plumbing system to the extent of implementing multiple, redundant check valves. Unfortunately, in nearly every trial, mobilization was observed to occur just after the transition from one pump to the other i.e. at a point of discontinuity in the smooth, regular, and gentle increase in flow velocity. Figure 2-25 shows the graphical overview of these trials and from the quantitative analysis of their results, it was obvious that this style of pump program was in fact less appropriate than the previous with a lower average critical velocity of 3.46 cm/s but larger 95% confidence interval of ± 0.45 cm/s, a larger standard deviation of 0.67 cm/s, and a larger coefficient of variation of 0.19.

Figure 2-25. Graphical summary of “reciprocating pumps” series of trial tests.

The outcome of this extensive period of testing (in total over 30 unique pump programs and almost 50 individual trials were performed) was the undeniable
acknowledgement that the incipient motion of a granular particle was indeed a very sensitive process whose characterization and quantification was precluded by the operational limitations of the syringe pumps. It was decided to abandon their use altogether and instead implement a system of constant head reservoirs as the fluid-driving system in the hopes of gaining better control of the flow behavior at incipient motion. Details of this phase of the research are presented next.

2.3 **Phase 3 – Constant Head Flow System & Flow Rate Sensor**

The third development phase of the experimental methodology included two major modifications: (1) upstream and downstream constant head reservoirs replaced the syringe pumps as the fluid driving system; and (2) an in-line flow sensor was implemented to directly measure the system’s flow rate. All other relevant aspects of the experiments remained unchanged. An overview of the experimental setup is shown in Figure 2-26.
Figure 2-26. Photographic overview of Phase 3 experimental setup with upstream and downstream constant head reservoirs and flow sensor.

### 2.3.1 Constant Head Flow System

In the Phase 3 experimental setup, fluid was provided to the flow cell by an upstream constant head reservoir while a second constant head reservoir received the flow cell’s effluent. Fluid was recirculated from the downstream reservoir back to the upstream reservoir with a submersible pump. The hydraulic head difference was controlled by raising or lowering a mechanical jack that supported the upstream reservoir, and additional flow control was gained through the use of a high precision needle valve (model: NV40025T, manufacturer: Hayward Flow Control) positioned upstream of the flow cell. Figure 2-27 shows the (a) upstream and (b) downstream constant head reservoirs (the upstream reservoir is partially submerged within a temperature control bath).
2.3.2 Inline Flow Sensor with Digital Readout

An inline flow sensor (model: FTB602B, manufacturer: Omega Engineering) with digital meter display (model: DPF701A, manufacturer: Omega Engineering) was installed between the outlet of the upstream reservoir and the flow control valve upstream of the flow cell. Although the intention was to use the sensor to measure the flow rate at incipient motion, unexplainable and data-corrupting problems with its operation ultimately led to its removal from the test setup.

Figure 2-27. Photographs showing (a) the upstream constant head reservoir in the temperature control bath and (b) the downstream reservoir sitting on the work bench.
2.3.3 Phase 3 Experimental Procedure and Method of Analysis

Test preparation for the Phase 3 trials remained unchanged from the method of Phase 2. The method of inducing the particle’s mobilization and determining the corresponding critical flow velocity, however, were markedly different. To reach the critical flow condition, the flow control valve was slowly opened to allow ever greater flow rates (and thus flow velocities) to reach the test particle. Manual observation of a test particle’s mobilization was made in real-time (with the aid of the camera and zoom lens) and marked the point at which further opening of the flow control valve ceased. The corresponding flow rate was referenced from the flow meter and taken to be the critical flow rate used to calculate the critical flow velocity.

2.3.4 Phase 3 Results and Identified Need for Improvements

During the process of validating the applicability of the new test methodology, a disqualifying (though difficult to detect) phenomenon was observed: the meter readout from the flow sensor would randomly and briefly display a sudden spike even when the levels of the constant head reservoirs and the valve were unchanged. This spike would last for only a split second before the readout would return to its normal low level of fluctuation. Two examples of this phenomenon are shown graphically in Figure 2-28. In these curves, the data for the flow rate was manually extracted from video recordings of the display meter and the method used to accomplish this extraction only allowed for the video’s elapsed time to be discretized into 1-s intervals. Thus, the actual duration of the
spikes and of the “normal” fluctuations in the meter’s output (which typically spanned intervals of less than whole seconds) are only approximated.

![Diagram showing flow rate/velocity spikes](image)

Figure 2-28. Estimated time series (extracted from video recording of flow meter display) highlighting flow rate/velocity spikes that appeared when using inline flow sensor.

In an attempt to identify the cause of these spikes, various components in the flow system were sequentially removed in the hopes that a process of elimination would reveal their source. Even after the system was reduced to a very simple form comprising the constant head reservoirs, the flow sensor, the tubing that conveyed the flow, and the hose barbs that connected the tubing to the other components, the appearance of the spikes persisted. Considering the critical requirement of an always-smooth change in flow rate (and thus flow velocity acting on the test particle) for the repeatable quantification of critical velocity, the decision was made to remove the flow sensor from the setup based on the rational that even the possibility of it being the source of the spikes was enough cause to justify its elimination. However, without the flow sensor or the prescribed flow rate from the pump programs, an alternative method of quantifying the flow rate was needed. Therefore, it was decided that a slight modification to the test setup would be
made to allow for the manual measurement of flow rate during each trial. Details of these modifications are discussed in the next section.

2.4 Phase 4 – Manual Flow Rate Measurement & Response Surface Methodology

The final development of the experimental methodology included three main modifications from the methodology of Phase 3: (1) a more sophisticated design of experiments (DOE) was developed; (2) the in-line flow sensor was eliminated and the downstream constant head reservoir was slightly modified to allow for manual measurements of the flow rate; and (3) the temperature control baths were implemented into the experimental setup. All other aspects of the experiments remained unchanged from the Phase 3 tests.

2.4.1 Improved Statistical Design and Implementation of Test Fluids

As highlighted by the observations and analyses of the results from the first three experimental phases, reliable measurement of the critical velocity proved to be rather sensitive to the effects of the testing environment (i.e. uncontrolled and/or lurking variables) and it became clear that the task of untangling these effects from those of the controlled variables (e.g. physicochemical fluid characteristics) could be aided by a more robust and sophisticated experimental design. It now seemed the original two-level full factorial design may have been an inefficient and inadequate approach to identifying the linear and nonlinear effects and interactions of experimental factors. As an alternative,
the experiments were redesigned using the concepts of the response surface methodology (RSM), which is known to be an effective and efficient method for the simultaneous estimation of the linear and quadratic effects as well as the interactions of the variables on the response parameters of interest (i.e. mean critical velocity and its standard deviation). Upon embarking on the redesign of the experiments, it was decided that suspended solids would be removed as a factor for two practical reasons: (1) in some earlier trial tests, it was discovered that maintaining the solids in suspension and avoiding clogging throughout the entire fluid system (e.g. the two constant head reservoirs, each with two separate compartments, the tubing with valves and hose barbs, the flow cell with flow distribution beads, etc) would be difficult if not impossible; and (2) in the new experimental design, using three factors instead of four significantly reduced the required number of tests.

The advantage of the RSM is that it allowed for the study of five distinct levels of each factor and the extraction of important statistical information from a relatively small number of experimental runs. For instance, a three-factor full factorial design of experiments with 5 levels for each factor requires $5^3=125$ experiments to be run (with no replication), while the RSM (as will be shown later) only requires 20 experiments and includes six replicates at the center point of the experiments.

There are two popular design approaches in RSM: the Box-Behken design and the central composite design (CCD). The latter approach was adopted in designing the experiments for the present study. The CCD, which was first introduced by Box and Wilson (1951), is obtained by combining three categories of data points; namely cubic, axial, and center points. An illustration of this three-dimensional experimental space for
the three test variables (i.e. viscosity: \( V \), pH: \( P \) and Ionic strength: \( I \)) investigated in this study is represented in Figure 2-29.

As shown in Figure 2-29, for \( k=3 \) variables, \( n_a=2k=6 \) axial points are defined in this design where one variable is set at its high or low level (i.e. the coded levels of \(-\alpha\) or \(+\alpha\)) while the other two variables are at intermediate levels (i.e. coded level of 0). As such, the axial points are the six points on the surface of the design pseudo-sphere where it intercepts each axis. The parameter \( \alpha \) is a number greater than one and equal to \( n_c^{1/4} \), where \( n_c=2^k (=8 \text{ for } k=3) \) is the number of cube points (Figure 2-29). Hence, the parameter \( \alpha \) in a 3-factor CCD will be equal to 1.682. In addition, six center points are collected at the origin (coded level of 0 for each of the three variables). Running six replicates is needed to attain a rotatable design (i.e. equal prediction variance for all points that are equidistant from the design center). The axial points along with the center points (which provide replication) enable the evaluation of linear and quadratic effects of the variables. The cube points (in which all variables are either at their intermediate-low or intermediate-high levels, coded respectively as \(-1\) and \(1\)) are the eight points at the vertices of the cube encircled by the design pseudo-sphere. These points enable the estimation of the two-way interactions between the variables. In total, 20 experiments will be necessary for estimating the linear and quadratic effects of the three variables as well as their two-way interactions.
In summary, if variable $X_i$ is to be studied over the range of $a$ to $b$ in the experiments (e.g. the pH is studied in the range of 3.5 to 10.5), these low and high levels are respectively coded as $-1.682$ and $+1.682$. The intermediate levels (associated with the coded level of 0) as well as the intermediate-high and intermediate-low levels (associated with the coded levels of $-1$ and $+1$, respectively) can be then calculated through linear interpolation. Using this approach and considering the studied ranges of the test variables, their five coded and natural levels can be computed as shown in Table 2-6. For each test variable, the coded factor levels of $-1.682$, $-1$, $0$, $+1$ and $+1.682$ are respectively labeled as low (L), intermediate-low (IL), intermediate (I), intermediate-high (IH) and high (H). The fluids are labeled by assigning the corresponding factor levels of viscosity, pH, and
ionic strength as the lowercase suffixes to V, P and I, respectively. For instance, $V_{IL}P_{IL}I_{IH}$ denotes a fluid having intermediate-low levels of viscosity and pH (i.e. 0.00697 g/cm·s and 4.92 g/cm·s, respectively), and an intermediate-high level of ionic strength (i.e. 0.24 mol/L).

Table 2-6. The Coded and Natural Levels of the Main Test Variables

<table>
<thead>
<tr>
<th></th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Low L (-α=-1.682)</td>
</tr>
<tr>
<td></td>
<td>Intermediate-Low IL (-1)</td>
</tr>
<tr>
<td></td>
<td>Intermediate I (0)</td>
</tr>
<tr>
<td></td>
<td>Intermediate-High IH (+1)</td>
</tr>
<tr>
<td></td>
<td>High H (+α=1.682)</td>
</tr>
<tr>
<td>Viscosity: V (g/cm·s)</td>
<td>0.00544</td>
</tr>
<tr>
<td></td>
<td>0.00697</td>
</tr>
<tr>
<td></td>
<td>0.00922</td>
</tr>
<tr>
<td></td>
<td>0.01147</td>
</tr>
<tr>
<td></td>
<td>0.013</td>
</tr>
<tr>
<td>pH: P</td>
<td>3.5</td>
</tr>
<tr>
<td></td>
<td>4.92</td>
</tr>
<tr>
<td></td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>9.08</td>
</tr>
<tr>
<td></td>
<td>10.50</td>
</tr>
<tr>
<td>Ionic strength: I (mol/L)</td>
<td>0.004</td>
</tr>
<tr>
<td></td>
<td>0.064</td>
</tr>
<tr>
<td></td>
<td>0.152</td>
</tr>
<tr>
<td></td>
<td>0.240</td>
</tr>
<tr>
<td></td>
<td>0.300</td>
</tr>
</tbody>
</table>

Table 2-7 shows the 20 designed experimental units (fluids) with their identifying labels along with the coded and natural levels of the test variables in a randomized run order.
Table 2-7. The Experimental Design with the Coded and Natural Levels of the Variables

<table>
<thead>
<tr>
<th>Run Order</th>
<th>Fluid ID</th>
<th>Viscosity: V (g/cm·s)</th>
<th>pH: P</th>
<th>Ionic Strength: I (mol/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Coded</td>
<td>Natural</td>
<td>Coded</td>
</tr>
<tr>
<td>14</td>
<td>V_{IL}P_{II}I_{IL}</td>
<td>-1</td>
<td>0.00697</td>
<td>-1</td>
</tr>
<tr>
<td>16</td>
<td>V_{II}P_{II}I_{II}</td>
<td>1</td>
<td>0.01147</td>
<td>-1</td>
</tr>
<tr>
<td>9</td>
<td>V_{IL}P_{II}I_{II}</td>
<td>-1</td>
<td>0.00697</td>
<td>1</td>
</tr>
<tr>
<td>12</td>
<td>V_{II}P_{III}I_{III}</td>
<td>1</td>
<td>0.01147</td>
<td>1</td>
</tr>
<tr>
<td>17</td>
<td>V_{IL}P_{II}I_{IH}</td>
<td>-1</td>
<td>0.00697</td>
<td>-1</td>
</tr>
<tr>
<td>5</td>
<td>V_{II}P_{II}I_{IH}</td>
<td>1</td>
<td>0.01147</td>
<td>-1</td>
</tr>
<tr>
<td>19</td>
<td>V_{IL}P_{III}I_{III}</td>
<td>-1</td>
<td>0.00697</td>
<td>1</td>
</tr>
<tr>
<td>13</td>
<td>V_{II}P_{III}I_{III}</td>
<td>1</td>
<td>0.01147</td>
<td>1</td>
</tr>
<tr>
<td>11</td>
<td>V_{II}P_{II}I</td>
<td>-1.682</td>
<td>0.00544</td>
<td>0</td>
</tr>
<tr>
<td>18</td>
<td>V_{II}P_{II}I</td>
<td>1.682</td>
<td>0.01300</td>
<td>0</td>
</tr>
<tr>
<td>4</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>-1.682</td>
</tr>
<tr>
<td>3</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>1.682</td>
</tr>
<tr>
<td>15</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>1</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>8</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
<tr>
<td>20</td>
<td>V_{II}P_{II}I</td>
<td>0</td>
<td>0.00922</td>
<td>0</td>
</tr>
</tbody>
</table>

Using the theory discussed previously in section 2.1.2, recipes were developed to guide the process of creating each fluid with its targeted factor levels (Table 2-8). The targeted temperatures were selected based on the assumption that each fluid would follow the well-established relationship between the temperature of distilled water and its corresponding viscosity, as described graphically in Figure 2-30 where the targeted low and high levels are also indicated.
Table 2-8. Recipes for Preparing the Experimental Fluids

<table>
<thead>
<tr>
<th>Run Order</th>
<th>Fluid ID</th>
<th>$T$ (°C)</th>
<th>Acid/base to Add</th>
<th>Acid/base for 11.5 L of Fluid (g)</th>
<th>NaCl for 11.5 L of Fluid (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
<tr>
<td>2</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
<tr>
<td>3</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>NaOH</td>
<td>0.1349</td>
<td>50.8795</td>
</tr>
<tr>
<td>4</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.3606</td>
<td>50.8640</td>
</tr>
<tr>
<td>5</td>
<td>V_{i}P_{i}I_{i}</td>
<td>14.63</td>
<td>HCl</td>
<td>0.0137</td>
<td>80.6394</td>
</tr>
<tr>
<td>6</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
<tr>
<td>7</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>100.8090</td>
</tr>
<tr>
<td>8</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
<tr>
<td>9</td>
<td>V_{i}P_{i}I_{i}</td>
<td>36.46</td>
<td>NaOH</td>
<td>0.0128</td>
<td>21.4869</td>
</tr>
<tr>
<td>10</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
<tr>
<td>11</td>
<td>V_{i}P_{i}I_{i}</td>
<td>50.00</td>
<td>NaOH</td>
<td>0.0002</td>
<td>51.0763</td>
</tr>
<tr>
<td>12</td>
<td>V_{i}P_{i}I_{i}</td>
<td>14.63</td>
<td>NaOH</td>
<td>0.0024</td>
<td>21.5020</td>
</tr>
<tr>
<td>13</td>
<td>V_{i}P_{i}I_{i}</td>
<td>14.63</td>
<td>NaOH</td>
<td>0.0024</td>
<td>80.6440</td>
</tr>
<tr>
<td>14</td>
<td>V_{i}P_{i}I_{i}</td>
<td>36.46</td>
<td>HCl</td>
<td>0.0137</td>
<td>21.4975</td>
</tr>
<tr>
<td>15</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>1.3441</td>
</tr>
<tr>
<td>16</td>
<td>V_{i}P_{i}I_{i}</td>
<td>14.63</td>
<td>HCl</td>
<td>0.0137</td>
<td>21.4975</td>
</tr>
<tr>
<td>17</td>
<td>V_{i}P_{i}I_{i}</td>
<td>36.46</td>
<td>HCl</td>
<td>0.0137</td>
<td>80.6394</td>
</tr>
<tr>
<td>18</td>
<td>V_{i}P_{i}I_{i}</td>
<td>9.76</td>
<td>HCl</td>
<td>0.0001</td>
<td>51.0765</td>
</tr>
<tr>
<td>19</td>
<td>V_{i}P_{i}I_{i}</td>
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<td>NaOH</td>
<td>0.0128</td>
<td>80.6289</td>
</tr>
<tr>
<td>20</td>
<td>V_{i}P_{i}I_{i}</td>
<td>23.89</td>
<td>HCl</td>
<td>0.0000</td>
<td>51.0766</td>
</tr>
</tbody>
</table>
2.4.2 The Regression Analysis of the Test Results

A regression analysis of the data obtained from the RSM can provide an approximate polynomial relationship between the response parameter of interest and the test variables per Equation (12). In this equation, \( y \) is the response parameter and \( X_i \) is the \( i^{th} \) test variable. \( X_i \cdot X_j \) is the interaction term between the \( i^{th} \) and \( j^{th} \) variable. A statistically significant \( \beta_{ij} \) indicates the presence of a meaningful interaction between these two terms (i.e. it influences the response). The term \( \varepsilon \) represents the residuals of the regression function, which is assumed to be a normal random variable with a mean of zero and standard deviation of \( \sigma \). The presence of the residual term is explained by the fact that the true relationship between the response and the variables is unknown and not necessarily polynomial. The proposed polynomial function will then cause some deviations in its predictions of the true results. The regression function for \( k=3 \) variables can be written per Equation (13). It is worth noting that while the proposed RSM design
is meant to be used for estimating up to the 2\textsuperscript{nd} order (i.e. quadratic) effects of the variables, the method can ultimately determine the cubic effects of the variables as well. Once the test data are analyzed to formulate the regression equation, the coefficients (i.e., $\beta_i$) can be estimated as $b_i$ (per Equation (14)) using the least sums of squares method. Unlike the true values of the coefficients ($\beta_i$) that are fixed and unknown, their estimates ($b_i$) are random variables with some mean and standard error. The ratio of the mean-to-standard error of these estimated coefficients will also be a random variable from a $t$-distribution. The significance of each term (which is equivalent to the significance of its coefficient) will be evaluated by calculating the probability of this ratio to be zero (which translates to insignificance of the term). This probability can be calculated using the cumulative distribution function of the $t$-distribution, and is denoted by the $p$-value. A significance level of 0.05 is commonly selected as the cut-off point for determining the significance of the regression terms (i.e. terms with $p$-values greater than 0.05 are deemed insignificant).

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i<j}^{k} \beta_{ij} X_i X_j + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \varepsilon$$ \hfill (12)

$$y = \beta_0 + \beta_1 \cdot X_1 + \beta_2 \cdot X_2 + \beta_3 \cdot X_3 + \beta_{12} \cdot X_1 \cdot X_2 + \beta_{13} \cdot X_1 \cdot X_3 + \beta_{23} \cdot X_2 \cdot X_3 + \beta_{11} \cdot X_1^2 + \beta_{22} \cdot X_2^2 + \beta_{33} \cdot X_3^2 + \varepsilon$$ \hfill (13)

$$y_i = b_0 + b_1 \cdot X_{1i} + b_2 \cdot X_{2i} + b_3 \cdot X_{3i} + b_{12} \cdot X_{1i} \cdot X_{2i} + b_{13} \cdot X_{1i} \cdot X_{3i} + b_{23} \cdot X_{2i} \cdot X_{3i} + b_{11} \cdot X_{1i}^2 + b_{22} \cdot X_{2i}^2 + b_{33} \cdot X_{3i}^2$$ \hfill (14)

The regression function obtained from Equation (14) can be used to predict the response parameters (at the experimented levels of the variables) whose deviations from
the actual observed values are the prediction errors. An adequate regression function will have minimal prediction errors. Such parameters are unbiased estimators of the residuals term \( (\varepsilon_i) \) and therefore must have a normal distribution with a generally constant variance. Tests to check the assumption of normality and constant variance must be performed to validate the assumptions of the regression analysis.

### 2.4.3 Phase 4 Experimental Procedure and Method of Analysis

The method of test preparation for the Phase 4 tests remained unchanged from the previous two phases. Two main alterations characterized the experimental procedure: (1) manual flow rate measurements; and (2) the use of temperature control baths.

The flow rate at incipient motion was determined manually by measuring the time required to collect a given volume of effluent. To facilitate this measurement, the tubing that previously exited from the flow cell and connected beneath the surface of the downstream constant head reservoir was relocated to hang over the top edge of the reservoir where it provided approximately 10 cm of tail head and allowed for the timed collections of effluent. The suitability of this method was confirmed by demonstrating its repeatability in several different trials. The results from this assessment are summarized in Table 2-9, which includes the raw data for the measurements of flow volume and time (note that the actual measurements were of the mass of effluent, which was then converted to volume based on the density as determined by the measured temperature), the calculated flow rates, and each trial’s average and standard deviation. It was concluded from the relatively low standard deviations in all cases that the method showed
good reproducibility and could be expected to contribute insignificantly to the overall variance in measuring the critical velocity.

To accurately control the fluid temperature (and thus, viscosity) both the upstream and downstream fluid reservoirs were placed in temperature control baths. The actual temperature of the test fluid in each trial (subsequently used to infer the fluid’s viscosity) was approximated based on the average of temperature measurements taken at the outlet of the upstream reservoir and within the main compartment of the downstream reservoir.
Table 2-9. Summary Table for Validative Assessment of Manual Flow Rate Measurements

<table>
<thead>
<tr>
<th>Trial</th>
<th>Effluent Collection Time (s)</th>
<th>Effluent Mass (g)</th>
<th>Effluent Temp. (°C)</th>
<th>Effluent Density (g/cm$^3$)</th>
<th>Effluent Volume (cm$^3$)</th>
<th>Flow Rate, Q (cm$^3$/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>306</td>
<td>35.24</td>
<td>326.25</td>
<td>23.08</td>
<td>0.9976</td>
<td>327.03</td>
<td>9.28</td>
</tr>
<tr>
<td></td>
<td>32.15</td>
<td>288.62</td>
<td>23.08</td>
<td>0.9976</td>
<td>289.31</td>
<td>9.00</td>
</tr>
<tr>
<td></td>
<td>37.53</td>
<td>334.52</td>
<td>23.15</td>
<td>0.9976</td>
<td>335.33</td>
<td>8.93</td>
</tr>
<tr>
<td></td>
<td>37.27</td>
<td>331.56</td>
<td>23.18</td>
<td>0.9976</td>
<td>332.37</td>
<td>8.92</td>
</tr>
<tr>
<td></td>
<td>41.13</td>
<td>362.88</td>
<td>23.24</td>
<td>0.9976</td>
<td>363.77</td>
<td>8.84</td>
</tr>
<tr>
<td></td>
<td>33.35</td>
<td>292.41</td>
<td>23.35</td>
<td>0.9975</td>
<td>293.13</td>
<td>8.79</td>
</tr>
<tr>
<td>307</td>
<td>30.29</td>
<td>352.54</td>
<td>23.55</td>
<td>0.9975</td>
<td>353.43</td>
<td>11.67</td>
</tr>
<tr>
<td></td>
<td>27.73</td>
<td>322.48</td>
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<td>0.9975</td>
<td>323.29</td>
<td>11.66</td>
</tr>
<tr>
<td></td>
<td>28.18</td>
<td>325.83</td>
<td>23.56</td>
<td>0.9975</td>
<td>326.65</td>
<td>11.59</td>
</tr>
<tr>
<td></td>
<td>28.53</td>
<td>331.74</td>
<td>23.55</td>
<td>0.9975</td>
<td>332.58</td>
<td>11.66</td>
</tr>
<tr>
<td></td>
<td>28.14</td>
<td>328.42</td>
<td>23.55</td>
<td>0.9975</td>
<td>329.25</td>
<td>11.70</td>
</tr>
<tr>
<td>308</td>
<td>29.44</td>
<td>320.40</td>
<td>23.55</td>
<td>0.9975</td>
<td>321.21</td>
<td>10.91</td>
</tr>
<tr>
<td></td>
<td>27.41</td>
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<td>23.54</td>
<td>0.9975</td>
<td>298.26</td>
<td>10.88</td>
</tr>
<tr>
<td></td>
<td>30.01</td>
<td>325.12</td>
<td>23.53</td>
<td>0.9975</td>
<td>325.94</td>
<td>10.86</td>
</tr>
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<td>309</td>
<td>31.84</td>
<td>339.92</td>
<td>23.49</td>
<td>0.9975</td>
<td>340.77</td>
<td>10.70</td>
</tr>
<tr>
<td></td>
<td>31.04</td>
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<td>23.52</td>
<td>0.9975</td>
<td>331.48</td>
<td>10.68</td>
</tr>
<tr>
<td>310</td>
<td>26.73</td>
<td>320.93</td>
<td>23.52</td>
<td>0.9975</td>
<td>321.74</td>
<td>12.04</td>
</tr>
<tr>
<td></td>
<td>26.47</td>
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<td>23.52</td>
<td>0.9975</td>
<td>317.95</td>
<td>12.01</td>
</tr>
<tr>
<td>311</td>
<td>27.37</td>
<td>330.27</td>
<td>23.54</td>
<td>0.9975</td>
<td>331.10</td>
<td>12.10</td>
</tr>
<tr>
<td></td>
<td>26.39</td>
<td>319.16</td>
<td>23.53</td>
<td>0.9975</td>
<td>319.96</td>
<td>12.12</td>
</tr>
<tr>
<td>312</td>
<td>26.37</td>
<td>287.90</td>
<td>23.56</td>
<td>0.9975</td>
<td>288.63</td>
<td>10.95</td>
</tr>
<tr>
<td></td>
<td>26.31</td>
<td>286.79</td>
<td>23.54</td>
<td>0.9975</td>
<td>287.51</td>
<td>10.93</td>
</tr>
<tr>
<td>313</td>
<td>26.39</td>
<td>310.76</td>
<td>23.60</td>
<td>0.9975</td>
<td>311.55</td>
<td>11.81</td>
</tr>
<tr>
<td></td>
<td>28.81</td>
<td>338.08</td>
<td>23.58</td>
<td>0.9975</td>
<td>338.93</td>
<td>11.76</td>
</tr>
<tr>
<td>314</td>
<td>26.16</td>
<td>327.46</td>
<td>23.59</td>
<td>0.9975</td>
<td>328.29</td>
<td>12.55</td>
</tr>
<tr>
<td></td>
<td>26.34</td>
<td>329.98</td>
<td>23.59</td>
<td>0.9975</td>
<td>330.81</td>
<td>12.56</td>
</tr>
</tbody>
</table>
Chapter 3

Experimental Results and Statistical Analyses

Table 3-1 presents a summary of the results from the 20 fluids tested in Phase 4 and includes the raw critical velocity data from each fluid’s seven repeat trials along with the 95% confidence intervals for their average and their standard deviation. Also shown for each fluid are its actual average temperature (measured at the time of testing) and standard deviation calculated from the seven repeats.

The observed critical velocities at particle incipient motion ($v_{cr}$) for the 20 fluids were analyzed with respect to the three fluid characteristics (i.e. factors/variables) ($\eta$, pH, $I$), and their individual and interactive influences on $v_{cr}$ were evaluated. The results suggest that viscosity ($\eta$), pH, and their two-way interaction are the most influencing parameters on the critical velocity at a granular particle’s incipient motion under laminar flow conditions. The ionic strength ($I$) was found to be of little effect on the critical velocity. Three different choices of variables and their combinations (known as product components) were selected and regression analyses were executed using the Minitab statistical software. It is noted that although many statistical tests are based on the assumption of a normally-distributed data set that simplifies the mathematical analysis, many real data sets do not strictly follow a normal distribution. However, an appropriate mathematical transformation of the raw data can often yield a comparable data set that does follow an approximately normal distribution. Such transformation increases the applicability and usefulness of statistical techniques based on the normality assumption.
The Box-Cox transformation (Box and Cox 1964) is a particularly useful family of transformations and is defined by Equation (15) where $Y$ is the response variable (in this case, the observed critical velocity) and $\lambda$ is the transformation parameter. If $\lambda = 0$, the natural log of the data is used instead.

$$T(Y) = \frac{Y^\lambda - 1}{\lambda}$$  \hspace{1cm} (15)

In each of the following five regression analyses, the Box-Cox transformation was first performed to transform the critical velocity values. The fluid characteristics ($\eta$, pH, $I$) and their combinations, referred to here as sources or predictors, were analyzed with respect to the transformed response. The first regression analysis contains the highest number of terms (predictors). Then, based on their relative significance, the insignificant variables were omitted in order to simplify the model and increase its predictive capability.
Table 3-1. Raw Critical Velocity Data and Summary Statistics

<table>
<thead>
<tr>
<th>Fluid ID</th>
<th>Critical Velocity, $v_c$ (cm/s)</th>
<th>95% CI for $\bar{x}<em>{vc}$ $s</em>{vc}$</th>
<th>$T \pm s_T$ (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>$V_{IP}I_I$</td>
<td>5.05</td>
<td>4.93</td>
<td>4.92</td>
</tr>
<tr>
<td>$V_{IP}I_I$</td>
<td>4.96</td>
<td>4.68</td>
<td>4.75</td>
</tr>
<tr>
<td>$V_{IPII}$</td>
<td>4.81</td>
<td>5.09</td>
<td>5.06</td>
</tr>
<tr>
<td>$V_{IPII}$</td>
<td>5.57</td>
<td>5.69</td>
<td>5.53</td>
</tr>
<tr>
<td>$V_{III}_I_I$</td>
<td>4.69</td>
<td>4.62</td>
<td>4.58</td>
</tr>
<tr>
<td>$V_{II}_I$</td>
<td>5.46</td>
<td>5.56</td>
<td>5.04</td>
</tr>
<tr>
<td>$V_{II}_H$</td>
<td>5.04</td>
<td>6.14</td>
<td>5.86</td>
</tr>
<tr>
<td>$V_{II}_I$</td>
<td>6.01</td>
<td>5.69</td>
<td>5.26</td>
</tr>
<tr>
<td>$V_{III}_II$</td>
<td>5.43</td>
<td>5.55</td>
<td>5.44</td>
</tr>
<tr>
<td>$V_{II}_I$</td>
<td>5.38</td>
<td>5.17</td>
<td>4.65</td>
</tr>
<tr>
<td>$V_{II}_H$</td>
<td>5.73</td>
<td>5.69</td>
<td>5.51</td>
</tr>
<tr>
<td>$V_{III}_II$</td>
<td>4.21</td>
<td>3.85</td>
<td>4.48</td>
</tr>
<tr>
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<td>4.29</td>
<td>4.29</td>
<td>4.22</td>
</tr>
<tr>
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<td>5.28</td>
<td>5.75</td>
<td>5.41</td>
</tr>
<tr>
<td>$V_{III}_II$</td>
<td>4.74</td>
<td>4.74</td>
<td>4.67</td>
</tr>
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<td>4.69</td>
<td>4.48</td>
<td>4.84</td>
</tr>
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<td>5.13</td>
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<td>4.38</td>
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<td>$V_{III}_II$</td>
<td>5.20</td>
<td>4.93</td>
<td>5.02</td>
</tr>
</tbody>
</table>

3.1 First Regression of $v_{cr}$ vs. $\eta$, pH, & $I$

Table 3-2 shows the analysis of variance table for the transformed response. The sources of variation included the regression function, each of the three fluid properties ($\eta$, pH, and $I$), all of their two-way interactions, and the error terms.
Table 3-2. Analysis of Variance Table for Transformed Response with Rounded $\lambda = -3$, estimated $\lambda = -3.15249$, 95% confidence interval for $\lambda$ is [-6.53299, 1.02101]

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Contribution</th>
<th>Adj SS</th>
<th>Seq MS</th>
<th>F-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
<td>9</td>
<td>5.09179</td>
<td>88.65%</td>
<td>5.09179</td>
<td>0.56575</td>
<td>8.68</td>
<td>0.001</td>
</tr>
<tr>
<td>$\eta$</td>
<td>1</td>
<td>3.96836</td>
<td>69.09%</td>
<td>0.16677</td>
<td>3.96836</td>
<td>60.87</td>
<td>0.000</td>
</tr>
<tr>
<td>pH</td>
<td>1</td>
<td>0.31136</td>
<td>5.42%</td>
<td>0.02194</td>
<td>0.31136</td>
<td>4.78</td>
<td>0.054</td>
</tr>
<tr>
<td>I</td>
<td>1</td>
<td>0.15738</td>
<td>2.74%</td>
<td>0.01191</td>
<td>0.15738</td>
<td>2.41</td>
<td>0.151</td>
</tr>
<tr>
<td>$\eta^*\eta$</td>
<td>1</td>
<td>0.20433</td>
<td>3.56%</td>
<td>0.21136</td>
<td>0.20433</td>
<td>3.13</td>
<td>0.107</td>
</tr>
<tr>
<td>pH*pH</td>
<td>1</td>
<td>0.00704</td>
<td>0.12%</td>
<td>0.00411</td>
<td>0.00704</td>
<td>0.11</td>
<td>0.749</td>
</tr>
<tr>
<td>I*pH</td>
<td>1</td>
<td>0.03789</td>
<td>0.66%</td>
<td>0.03789</td>
<td>0.03789</td>
<td>0.58</td>
<td>0.463</td>
</tr>
<tr>
<td>$\eta^*pH$</td>
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<td>0.30438</td>
<td>5.30%</td>
<td>0.30438</td>
<td>0.30438</td>
<td>4.67</td>
<td>0.056</td>
</tr>
<tr>
<td>$\eta^*I$</td>
<td>1</td>
<td>0.01203</td>
<td>0.21%</td>
<td>0.01203</td>
<td>0.01203</td>
<td>0.18</td>
<td>0.677</td>
</tr>
<tr>
<td>pH*I</td>
<td>1</td>
<td>0.08903</td>
<td>1.55%</td>
<td>0.08903</td>
<td>0.08903</td>
<td>1.37</td>
<td>0.270</td>
</tr>
<tr>
<td>Error</td>
<td>10</td>
<td>0.65192</td>
<td>11.35%</td>
<td>0.65192</td>
<td>0.65192</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Lack-of-Fit</td>
<td>5</td>
<td>0.45481</td>
<td>7.92%</td>
<td>0.45481</td>
<td>0.09096</td>
<td>2.31</td>
<td>0.190</td>
</tr>
<tr>
<td>Pure Error</td>
<td>5</td>
<td>0.19711</td>
<td>3.43%</td>
<td>0.19711</td>
<td>0.03942</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>5.74371</td>
<td>100.00%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

The table columns are defined as follows: **Source** = all possible sources that contribute to variation of the observations from the fitted explanatory model; **DF** = degree of freedom; **Seq SS** = sequential sum of squared variations in the response (that is $\nu_{cr}$) due to the variation in each source; **Contribution** = relative contribution of each source to the total variation in the response, it is calculated by dividing the Seq SS of each source by the total term; **Seq MS** = sequential mean of the squared variations, it is the Seq SS divided by the degrees of freedom and the greater its value compared to the Error term, the more significant the source is in explaining the response; **F-Value** = Seq SS of each source divided by the Seq SS of the Error term and the greater its value, the more significant the source is in explaining the response; **p-value** = the probability of rejecting the hypothesis that the source is significant. For example, in this table, the p-
value is 0.054 for viscosity, meaning the probability of mistakenly concluding that pH has a significant effect on the response is 0.054. In addition, the regression analysis reports a p-value for “lack-of-fit”, which, if less than 0.05, suggests the proposed model is incapable of explaining the variations in the response. In this regression analysis, the p-value for “lack-of-fit” is 0.190, indicating the regression model is capable of capturing the variations in the response.

The significance of the effects of viscosity, pH, and ionic strength and their interactive effects on the critical velocity for a particle’s incipient motion can be judged by the p-values in Table 3-2. This analysis suggests that at a level of confidence of 95%, viscosity has a strongly significant effect on critical velocity and pH has a marginally significant effect.

The regression analysis produced the following model summary for the transformed response: $R^2 = 88.65\%$, $R^2_{\text{adj}} = 78.43\%$, and $R^2_{\text{pred}} = 35.06\%$. $R^2$ is coefficient of determination and the greater its value, the better the model fits the available data. However, $R^2$ is often criticized since its value increases when more terms or predictors are added to the regression function, which actually decreases the predictive capability of the model. Therefore, two other types of $R^2$ are defined that address the deficiency of the conventional $R^2$, namely the adjusted $R^2$ (defined as $R^2_{\text{adj}}$) and the predicted $R^2$ (defined as $R^2_{\text{pred}}$). The adjusted $R^2$ penalizes the regression function for its over-parametrization (i.e. adding more terms decreases its value). Therefore, the greater the number of insignificant terms that are included in the model, the more the adjusted $R^2$ drops from its conventional value. The predicted $R^2$ is regarded the best criterion for assessing the goodness of fit and predictive capabilities of the model.
In order to derive a regression equation that quantifies the effect of each predictor on the response, Table 3-3 provides the coefficients for the transformed responses. In this table, Coef is the coefficient for each source in the derived regression equation for the response or transformation of the response. Positive coefficients have a reducing effect on the response, while negative coefficients have an increasing effect.

Table 3-3. Coefficients for Transformed Responses

<table>
<thead>
<tr>
<th>Term</th>
<th>Coef</th>
<th>SE Coef</th>
<th>95% CI</th>
<th>T-Value</th>
<th>P-Value</th>
<th>VIF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>212.23</td>
<td>2.08</td>
<td>(207.61, 216.86)</td>
<td>102.21</td>
<td>0.000</td>
<td>-</td>
</tr>
<tr>
<td>η</td>
<td>46.4</td>
<td>29.0</td>
<td>(-18.3, 111.1)</td>
<td>1.60</td>
<td>0.141</td>
<td>89.19</td>
</tr>
<tr>
<td>pH</td>
<td>0.170</td>
<td>0.293</td>
<td>(-0.482, 0.822)</td>
<td>0.58</td>
<td>0.575</td>
<td>77.71</td>
</tr>
<tr>
<td>I</td>
<td>-2.61</td>
<td>6.10</td>
<td>(-16.20, 10.98)</td>
<td>-0.43</td>
<td>0.678</td>
<td>60.35</td>
</tr>
<tr>
<td>η*η</td>
<td>-240</td>
<td>133</td>
<td>(-536, 57)</td>
<td>-1.80</td>
<td>0.102</td>
<td>64.81</td>
</tr>
<tr>
<td>pH*pH</td>
<td>0.0039</td>
<td>0.0155</td>
<td>(-0.0307, 0.0385)</td>
<td>0.25</td>
<td>0.807</td>
<td>43.90</td>
</tr>
<tr>
<td>I*I</td>
<td>-6.62</td>
<td>8.68</td>
<td>(-25.97, 12.73)</td>
<td>-0.76</td>
<td>0.463</td>
<td>12.33</td>
</tr>
<tr>
<td>η*pH</td>
<td>-4.17</td>
<td>1.93</td>
<td>(-8.47, 0.13)</td>
<td>-2.16</td>
<td>0.056</td>
<td>49.04</td>
</tr>
<tr>
<td>η*I</td>
<td>19.6</td>
<td>45.6</td>
<td>(-82.1, 121.3)</td>
<td>0.43</td>
<td>0.677</td>
<td>34.82</td>
</tr>
<tr>
<td>pH*I</td>
<td>0.576</td>
<td>0.493</td>
<td>(-0.522, 1.674)</td>
<td>1.17</td>
<td>0.270</td>
<td>25.41</td>
</tr>
</tbody>
</table>

Equation (16) shows the regression equation obtained from Table 3-3 and suggests that increased viscosity, increased pH, and decreased ionic strength have a decreasing effect on critical velocity. This is in agreement with the findings of previous studies (Arulanandan et al. 1975; Hubbe 1985; Reddi et al. 2000a; Sharma et al. 1992; Sherard et al. 1972) as discussed in Chapter 1.

\[
\frac{\nu_{cr}^{\lambda-1}}{\lambda \cdot g^{\lambda-1}} = 212.23 + 46.4\eta + 0.17pH - 2.61I - 240\eta \cdot \eta + 0.0039pH \cdot pH - 6.62I \cdot I \\
- 4.17\eta \cdot pH + 19.6\eta \cdot I + 0.576pH \cdot I
\]  

(16)

Where \( \lambda = -3 \)
\[ g = 5.04042 = \text{geometric mean of } v_{cr} \]

### 3.2 Second (reduced) Regression of \( v_{cr} \) vs. \( \eta, \text{pH, } \text{& } I \)

In this regression analysis, the higher order terms (sources) that were found to be insignificant to the response in the first regression (i.e. \( \eta^*\eta, \text{pH*pH, } I*I, \eta*I, \text{pH*I} \)) were excluded. Only the three main factors (\( \eta, \text{pH, } I \)) and the interaction of viscosity and pH were included. Accordingly, the Box-Cox transformation constant (\( \lambda \)) changed since the sources (predictors) changed. Table 3-4 shows the analysis of variance table for the transformed responses. Similar to the outcome of the first regression, at a level of confidence of 95%, both viscosity and pH were shown to be significant while ionic strength remained insignificant.

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Contribution</th>
<th>Adj SS</th>
<th>Seq MS</th>
<th>F-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
<td>4</td>
<td>0.006449</td>
<td>81.89%</td>
<td>0.006449</td>
<td>0.001612</td>
<td>16.95</td>
<td>0.000</td>
</tr>
<tr>
<td>( \eta )</td>
<td>1</td>
<td>0.005490</td>
<td>69.71%</td>
<td>0.000003</td>
<td>0.00549</td>
<td>57.73</td>
<td>0.000</td>
</tr>
<tr>
<td>pH</td>
<td>1</td>
<td>0.000384</td>
<td>4.88%</td>
<td>0.000218</td>
<td>0.000384</td>
<td>4.04</td>
<td>0.063</td>
</tr>
<tr>
<td>I</td>
<td>1</td>
<td>0.000227</td>
<td>2.88%</td>
<td>0.000227</td>
<td>0.000227</td>
<td>2.38</td>
<td>0.143</td>
</tr>
<tr>
<td>( \eta^*\text{pH} )</td>
<td>1</td>
<td>0.000349</td>
<td>4.43%</td>
<td>0.000349</td>
<td>0.000349</td>
<td>3.67</td>
<td>0.075</td>
</tr>
<tr>
<td>Error</td>
<td>15</td>
<td>0.001426</td>
<td>18.11%</td>
<td>0.001426</td>
<td>0.000095</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Lack-of-Fit</td>
<td>10</td>
<td>0.001103</td>
<td>14.01%</td>
<td>0.001103</td>
<td>0.001103</td>
<td>1.71</td>
<td>0.289</td>
</tr>
<tr>
<td>Pure Error</td>
<td>5</td>
<td>0.000323</td>
<td>4.10%</td>
<td>0.000323</td>
<td>0.000065</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>0.007876</td>
<td>100.00%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 3-4. Analysis of Variance Table for Transformed Response where Rounded \( \lambda = -1 \), estimated \( \lambda = -0.7793 \), 95% confidence interval for \( \lambda \) is \([-4.09580, 2.88820]\).

The reduced regression analysis produced the following model summary for the transformed response: \( R^2 = 81.89\%, R_{adj}^2 = 77.06\%, \) and \( R_{pred}^2 = 64.33\% \). It is noted that
the predictive $R^2$ value increased from 35.06% in the first regression to 64.33% in this analysis, indicating an increase in the prediction power when insignificant factors are excluded. The results still suggest that ionic strength has an insignificant effect on the response.

Table 3-5 lists the coefficients for the transformed responses that produced the following regression equation:

$$-\frac{v_{cr}}{v} = -0.1974 + 0.096\eta + 0.01047\text{pH} + 0.0463I - 0.1412\eta \cdot \text{pH}$$  \hspace{1cm} (17)

Again, similar to the first regression, the equation shows increased viscosity and pH decrease the critical velocity, while ionic strength remains insignificant.

<table>
<thead>
<tr>
<th>Term</th>
<th>Coef</th>
<th>SE Coef</th>
<th>95% CI</th>
<th>T-Value</th>
<th>P-Value</th>
<th>VIF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-0.1974</td>
<td>0.0498</td>
<td>(-0.3037, -0.0912)</td>
<td>-3.96</td>
<td>0.001</td>
<td>-</td>
</tr>
<tr>
<td>$\eta$</td>
<td>0.096</td>
<td>0.529</td>
<td>(-1.032, 1.224)</td>
<td>0.18</td>
<td>0.858</td>
<td>20.31</td>
</tr>
<tr>
<td>pH</td>
<td>0.01047</td>
<td>0.00691</td>
<td>(-0.00427, 0.02520)</td>
<td>1.51</td>
<td>0.151</td>
<td>29.73</td>
</tr>
<tr>
<td>I</td>
<td>0.0463</td>
<td>0.0300</td>
<td>(-0.0176, 0.1102)</td>
<td>1.54</td>
<td>0.143</td>
<td>1.00</td>
</tr>
<tr>
<td>$\eta \cdot \text{pH}$</td>
<td>-0.1412</td>
<td>0.0737</td>
<td>(-0.2983, 0.0160)</td>
<td>-1.91</td>
<td>0.075</td>
<td>49.04</td>
</tr>
</tbody>
</table>

3.3 Regression of $v_{cr}$ vs. $\eta$, pH with 0.05 Significance Level

In this regression analysis, ionic strength was excluded since it was shown to be insignificant in both of the previous analyses. Only viscosity, pH and their interaction ($\eta \cdot \text{pH}$) were included in the analysis. Table 3-6 shows the analysis of variance table for the transformed response. The P-values for pH and $\eta \cdot \text{pH}$ are 0.078 and 0.094, respectively. The table shows there are unknown (lurking) variables or some other disturbances (statistical artifacts) that blur the effects of the main variables, as indicated
by the contribution of 21.39% from the Error term. By lowering the level of confidence from 95% to 90%, pH and the interaction of pH and viscosity can be included as significant terms. This final regression will be shown in the following section.

Table 3-6. Analysis of Variance Table for Transformed Response where Rounded $\lambda = -0.5$, estimated $\lambda = -0.703655$, 95% confidence interval for $\lambda$ is [-4.15116, 3.11084]

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Contribution</th>
<th>Adj SS</th>
<th>Seq MS</th>
<th>F-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
<td>3</td>
<td>0.007651</td>
<td>78.61%</td>
<td>0.007651</td>
<td>0.00255</td>
<td>19.6</td>
<td>0.000</td>
</tr>
<tr>
<td>$\eta$</td>
<td>1</td>
<td>0.006775</td>
<td>69.62%</td>
<td>0.000002</td>
<td>0.006775</td>
<td>52.08</td>
<td>0.000</td>
</tr>
<tr>
<td>pH</td>
<td>1</td>
<td>0.000462</td>
<td>4.75%</td>
<td>0.000257</td>
<td>0.000462</td>
<td>3.55</td>
<td>0.078</td>
</tr>
<tr>
<td>$\eta$*pH</td>
<td>1</td>
<td>0.000413</td>
<td>4.25%</td>
<td>0.000413</td>
<td>0.000413</td>
<td>3.18</td>
<td>0.094</td>
</tr>
<tr>
<td>Error</td>
<td>16</td>
<td>0.002082</td>
<td>21.39%</td>
<td>0.002082</td>
<td>0.00013</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Lack-of-Fit</td>
<td>11</td>
<td>0.001668</td>
<td>17.14%</td>
<td>0.001668</td>
<td>0.000152</td>
<td>1.83</td>
<td>0.261</td>
</tr>
<tr>
<td>Pure Error</td>
<td>5</td>
<td>0.000413</td>
<td>4.25%</td>
<td>0.000413</td>
<td>0.000083</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>0.009732</td>
<td>100.00%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

This regression analysis yielded the following model summary for the transformed response: $R^2 = 78.61\%$, $R^2_{\text{adj}} = 74.60\%$, and $R^2_{\text{pred}} = 66.60\%$. Compared with the preceding analysis, only a slight increase in the predictive capability of the model was achieved in this regression. Table 3-7 lists the coefficients for transformed responses that were used to generate the regression function (Equation 18).

\[-v_{cr}^{0.5} = -0.4342 + 0.084\eta + 0.01137\text{pH} - 0.1536\eta \cdot \text{pH}\]  \hspace{1cm} (18)

Table 3-7. Coefficients for Transformed Responses

<table>
<thead>
<tr>
<th>Term</th>
<th>Coef</th>
<th>SE Coef</th>
<th>95% CI</th>
<th>T-Value</th>
<th>P-Value</th>
<th>VIF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-0.4342</td>
<td>0.0581</td>
<td>(-0.5573, -0.3111)</td>
<td>-7.48</td>
<td>0.000</td>
<td>-</td>
</tr>
<tr>
<td>$\eta$</td>
<td>0.084</td>
<td>0.619</td>
<td>(-1.228, 1.397)</td>
<td>0.14</td>
<td>0.893</td>
<td>20.31</td>
</tr>
<tr>
<td>pH</td>
<td>0.01137</td>
<td>0.00809</td>
<td>(-0.00577, 0.02851)</td>
<td>1.41</td>
<td>0.179</td>
<td>29.73</td>
</tr>
<tr>
<td>$\eta$*pH</td>
<td>-0.1536</td>
<td>0.0862</td>
<td>(-0.3364, 0.0291)</td>
<td>-1.78</td>
<td>0.094</td>
<td>49.04</td>
</tr>
</tbody>
</table>
3.4 Regression of $v_{cr}$ vs. $\eta$, pH with 0.1 Significance Level

In this regression analysis, the response factors from the previous regression analysis (section 3.3) were maintained and the level of confidence was reduced from 95% to 90%. As such, the only significant change in the outcome is in the width of the confidence interval for the transformed responses (Table 3-8) and in the perspective from which the p-values in the analysis of variance table are interpreted. In other words, from the new perspective, any p-value less than 0.1 (rather than the previous cutoff of 0.05) is considered significant. Therefore, the three terms (viscosity, pH, and their interaction) are now considered significant at a level of confidence of 90%.

Table 3-8. Coefficients for Transformed Response

<table>
<thead>
<tr>
<th>Term</th>
<th>Coef</th>
<th>SE Coef</th>
<th>90% CI</th>
<th>T-Value</th>
<th>P-Value</th>
<th>VIF</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-0.4342</td>
<td>0.0581</td>
<td>(-0.5355, -0.3328)</td>
<td>-7.48</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>$\eta$</td>
<td>0.084</td>
<td>0.619</td>
<td>(-0.996, 1.165)</td>
<td>0.14</td>
<td>0.893</td>
<td>20.31</td>
</tr>
<tr>
<td>pH</td>
<td>0.01137</td>
<td>0.00809</td>
<td>(-0.00275, 0.02549)</td>
<td>1.41</td>
<td>0.179</td>
<td>29.73</td>
</tr>
<tr>
<td>$\eta \times$pH</td>
<td>-0.1536</td>
<td>0.0862</td>
<td>(-0.3042, -0.0031)</td>
<td>-1.78</td>
<td>0.094</td>
<td>49.04</td>
</tr>
</tbody>
</table>

3.5 Model Validation and Interpretation of Effects

The residuals plots in Figure 3-1 check the validity of the assumptions made in performing the regression analyses. The plot of the ordered residuals (bottom right) shows how the residuals of the predictions of the regression model changed between consecutive observations and shows no invalidating systematic trend. In the top left plot, the close fit of the individual observations to the normal curve validate the normality of the data set. The plot on the top right tests the consistency of the residuals’ variance
where a regular spread from the zero line throughout the range of fitted values is desired. It suggests there may be some some issues with inconsistent variance between experimental units (i.e. test fluids). Finally, the histogram in the bottom left is another check on the normality of the data and shows no disqualifying characteristics.

Figure 3-1. Residuals plots for critical velocity.

Figure 3-2 shows how the mean response of critical velocity changed as the significant variables changed across their studied ranges. This figure quantitatively shows the relative effects of viscosity and pH on the particle’s incipient motion: the variation of \( v_{cr} \) ranged from approximately 4.25 cm/sec to 6.0 cm/sec when the viscosity changed from 0.013 (g/cm·s) to 0.0055 (g/cm·s) while \( v_{cr} \) ranged from approximately from 4.8 cm/sec to 5.25 cm/sec when the pH changed from 10.5 to 3.5.
Figure 3-3 shows the effect on mean critical viscosity caused by the interaction between viscosity and pH. When the pH was higher (at 10.5) the viscosity had a greater effect on the critical velocity than when the pH was lower (at 3.5), as indicated by the greater slope of the line for the higher pH.

Figure 3-4 shows the contour plot of the critical velocity responses to viscosity and pH. The plot reveals the same observation as in Figure 3-3: at a higher pH, the effect of viscosity on critical velocity was more pronounced than at a lower pH. The plot also reveals the same conclusion that was derived from Tables 3-6 to 3-8: viscosity affected the critical velocity more than pH. In addition, at a viscosity of approximately 0.007 g/cm·s, the critical velocity remained relatively unchanged as pH increased from 3.5 to 10.5. At a viscosity greater than 0.007 g/cm·s, the critical velocity decreased with
increasing pH. At a viscosity less than 0.007 g/cm·s, the effect of pH on the critical velocity reversed: the critical velocity increased with an increase in pH. At any pH value (if the contour lines are extrapolated), critical velocity decreases with the increase of viscosity.

Figure 3-3. Effect of the interaction between viscosity and pH on critical velocity.
Figures 3-4, 5 and 3-6 are two optimization plots that detect the variable combinations that yield the highest and lowest critical velocity, respectively. In the test setup employed in this research, the lowest critical velocity occurred in the fluid with $\eta = 0.013$ g/cm·s (the highest viscosity used in all the fluids) and pH = 7.88 (an intermediate pH value of those used in all the fluids); the highest critical velocity occurred in the fluid with $\eta = 0.00544$ g/cm·s (the lowest viscosity used in all the fluids) and pH = 4.77 (an intermediate-low pH value of those used in all the fluids).
Figure 3-5. Optimization plot detecting the variable combination that yielded the lowest critical velocity.

<table>
<thead>
<tr>
<th>Optimal</th>
<th>η</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>D: 1.000</td>
<td>0.013</td>
<td>10.50</td>
</tr>
<tr>
<td>Predict</td>
<td>Cur</td>
<td>0.013</td>
</tr>
<tr>
<td>Low</td>
<td>0.00544</td>
<td>3.50</td>
</tr>
</tbody>
</table>

Vc
Minimum
y = 4.1476
d = 1.0000

critical velocity.

Figure 3-6. Optimization plot detecting the variable combination that yielded the highest critical velocity.

<table>
<thead>
<tr>
<th>Optimal</th>
<th>η</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>D: 1.000</td>
<td>0.013</td>
<td>10.50</td>
</tr>
<tr>
<td>Predict</td>
<td>Cur</td>
<td>0.013</td>
</tr>
<tr>
<td>Low</td>
<td>0.00544</td>
<td>3.50</td>
</tr>
</tbody>
</table>

Vc
Maximum
y = 5.8006
d = 1.0000
3.6 Regression Analysis of the Standard Deviations of the Sample Means of the Observed Critical Velocity Values

An additional regression analysis was performed on the standard deviations of the sample means of the observed critical velocity values in the 20 fluids; the sources (predictors) included viscosity, pH, ionic strength, the interaction of viscosity and pH, and the error terms. The purpose of this analysis was to identify the factors that contributed most to the variation of the response (critical velocity) in each of the 20 fluids. Table 3-9 shows the analysis of variance table for the transformed response, where only 34.71% of the observed variations were associated with the test variables and 65.29% of the variations were due to unexplained errors. The table also shows the ionic strength and the interaction of the pH and viscosity as the only parameters that could marginally explain part of the reason for the different variations in the \( v_{cr} \) results of the different fluids.

Table 3-9. Analysis of Variance Table for Transformed Response where Rounded \( \lambda = 0 \), estimated \( \lambda = 0.0811278 \), 95% confidence interval for \( \lambda \) is [-0.753372, 0.910628]

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
<th>Contribution</th>
<th>Adj SS</th>
<th>Seq MS</th>
<th>F-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
<td>4</td>
<td>1.61521</td>
<td>34.71%</td>
<td>1.6152</td>
<td>0.403802</td>
<td>1.99</td>
<td>0.147</td>
</tr>
<tr>
<td>( \eta )</td>
<td>1</td>
<td>0.26777</td>
<td>5.75%</td>
<td>1.0269</td>
<td>0.267766</td>
<td>1.32</td>
<td>0.268</td>
</tr>
<tr>
<td>pH</td>
<td>1</td>
<td>0.00078</td>
<td>0.02%</td>
<td>0.8114</td>
<td>0.000778</td>
<td>0</td>
<td>0.951</td>
</tr>
<tr>
<td>I</td>
<td>1</td>
<td>0.49749</td>
<td>10.69%</td>
<td>0.4975</td>
<td>0.49749</td>
<td>2.46</td>
<td>0.138</td>
</tr>
<tr>
<td>( \eta*pH )</td>
<td>1</td>
<td>0.84917</td>
<td>18.25%</td>
<td>0.8492</td>
<td>0.849174</td>
<td>4.19</td>
<td>0.059</td>
</tr>
<tr>
<td>Error</td>
<td>15</td>
<td>3.03867</td>
<td>65.29%</td>
<td>3.0387</td>
<td>0.202578</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Lack-of-Fit</td>
<td>10</td>
<td>1.37400</td>
<td>29.52%</td>
<td>1.3740</td>
<td>0.137400</td>
<td>0.41</td>
<td>0.89</td>
</tr>
<tr>
<td>Pure Error</td>
<td>5</td>
<td>1.66466</td>
<td>35.77%</td>
<td>1.6647</td>
<td>0.332932</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>19</td>
<td>4.65387</td>
<td>100.00%</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Figure 3-7 shows how the standard deviation of the sample means of critical velocity ($sV_c$) was affected by each variable (i.e., viscosity, pH, ionic strength). The figure again shows viscosity and ionic strength were the main contributors to the variation of the test results while pH was of little impact.

![Main Effects Plot for $sV_c$]

Figure 3-7. Contributions of viscosity, pH, and ionic strength to the standard deviation of critical velocity sample means.

Because the interaction of viscosity and pH was identified a significant factor, this term was also analyzed for its contribution to the standard deviation of the sample means of critical velocity; the results are shown in Figure 3-8. At a pH value of 3.5, increased viscosity decreased the variation of the test results; at a pH value of 10.5, increased viscosity increased the variation of the test results. It can be concluded that fluids with high viscosity, low pH and low ionic strength cause low variations in critical velocity...
while fluids with low viscosity, low pH and high ionic strength cause high variations in critical velocity.

![Interaction Plot for sVc](image)

Figure 3-8. Contributions of viscosity–pH interaction to the standard deviation of critical velocity.

3.7 Practical Limitations and Suspected Major Sources of Variation

In light of the preceding analyses, two main sources of variation were suspected: (1) changes in the resistance to erosion offered by the specific orientation of the test particle and the support domes; and (2) the manner in which the increasing flow intensity was imparted upon the test particles.

During the course of conducting the Phase 4 experiments, it was observed that the test particle could reside in a quasi-stable state, and ultimately become mobilized from, more than one location atop the domes. In other words, the initial intent of providing
three consistent and repeatable points of contact to support the test particle at incipient motion was not fully achieved. A composite image of three photographs illustrates this behavior in Figure 3-9. In each of the three photographs used to create the composite image, the test particle resided at a different (but stable, due to the force imparted upon it by the flow) position atop the support domes. To aid in distinguishing the three particle/positions from one another, arrows and labels have been added to demarcate the leading and trailing edges. It was qualitatively observed throughout the testing program that the particle typically became mobilized from either of the two most downstream positions highlighted in the image, though attempts to repeatedly achieve one or the other were ultimately unsuccessful. It was suspected that each position afforded the test particle a slightly different resistance to erosion and by virtue of their relation, affected the perceived critical velocity and inflated the variation in the results.

The other suspected major source of variation was the needle valve and the fact that it was operated manually. With the flow rate and general flow conditions being such important factors in the experiments, a more repeatable and consistent method of introducing and adjusting the flow intensity could be beneficial. Many of the experimental trials were abandoned due to the fact that the valve was opened too quickly or abruptly and provoked what was assumed to be a premature mobilization at an exceptionally low flow velocity. On these instances, the result was simply frustration on the part of the experimenter. However, on other occasions, for example at flow velocities closer to the perceived critical velocity, such an event could mistakenly be recorded as an actual measurement of the critical velocity and thus add to the variance of the response.
Figure 3-9. Composite image of three photographs showing multiple test particle positions atop support domes.
Chapter 4

Analytical Study of the Forces on the Test Particle under the Effects of Fluid Viscosity, pH, and Ionic Strength

This chapter analytically investigates the roles of a fluid’s viscosity, pH, and ionic strength in the force equilibrium of a granular particle on a particle bed. The purpose of this analytical study is to provide a theoretical explanation for the observed effects these factors had on the mobilization of the test particle, as presented in Chapter 3. It is shown that viscosity affects the mobilizing hydraulic shear force, while pH and ionic strength affect the surface forces between the particle and the particle bed.

4.1 Hydrodynamic Force Acting on a Submerged Particle

The hydrodynamic forces include shear force and lift force. The tangential (also known as horizontal/shear) force on a spherical particle of radius \( R \) in contact with a plane in a slow linear shear flow (i.e., Couette flow) was provided by Goldman et al. (1967) and O’Neill (1968):

\[
F_H = 1.7(6\pi) \mu R u_x
\]  

(19)

Where \( \mu \) = dynamic viscosity

\( R \) = particle radius

\( u_x \) = the flow velocity in the \( x \) (horizontal) direction at a distance \( R \) measured from the plane on which the particle rests.
In this research, the effect of the three support domes on \( u_x \) in the experimental program was neglected. The flow condition was assumed to be fully submerged flow between two parallel plates, as described in Section 2.1.3 and shown in Figure 2-4. The parabolic flow velocity \( (u) \) profile of pressure-driven Poiseuille flow between two parallel plates can be expressed by Equation (9).

At the center of the test particle, \( y = h/2 - R \). Therefore, the velocity at the center of the particle, \( u_p \), can be expressed as:

\[
   u_p = 6 \left( \frac{Q}{A} \right) \left( \frac{R}{h} \right) \left( 1 - \frac{R}{h} \right) = 6u_{ave} \left( \frac{R}{h} \right) \left( 1 - \frac{R}{h} \right)
\]

(21)

Where

\[
   Q = \text{flow rate}
\]

\[
   A = \text{flow cross-sectional area}
\]

\[
   u_{ave} = \frac{Q}{A}
\]

In this study, \( h = 2.6 \times 10^{-3} \text{ m} \), and \( R = 0.345 \times 10^{-3} \text{ m} \). Based on the experimental program, the critical average flow velocity for the 20 test fluids ranges from 4.16±0.24 cm/s to 5.80±0.27 cm/s, so the maximum range of the critical average flow velocity is [3.92 cm/s, 6.07 cm/s]. In this study, viscosity ranges from 0.000544 N·s/m² to 0.001317 N·s/m². The computed variation of hydraulic shear force \( (F_H) \) with dynamic viscosity is shown in Figure 4-1.
4.2 Inter-particle Surface Forces

The inter-particle surface forces between the test particle and the support domes include the London-van der Waals force (negative, attraction force) and the electrostatic force (positive, repulsive force). The net surface force acting on the particle is the sum of these opposing forces.

\[ P_{\text{net surface force}} = P_{\text{van der Waals}} + P_{\text{electrostatic}} \] (22)
4.2.1 van der Waals Force

The van der Waals force between two spheres (as illustrated in Figure 4-2) can be expressed by (Israelachvili 2011):

\[ p_{\text{van der Waals}} = -\frac{A}{6D^2}\left(\frac{R_1R_2}{R_1 + R_2}\right) \]

(23)

Where

- \( A \) = the Hamaker constant
- \( D \) = the particle separation distance
- \( R_1 \) = the radius of the test particle = 0.345 mm
- \( R_2 \) = the radii of the support domes = 0.188 mm

![Diagram of two spheres with separation distance D.](image)

Figure 4-2. Illustration of two spheres with separation distance \( D \).

The Hamaker constant can vary from \( 1.05 \times 10^{-13} \) ergs for glass-water-glass contact to \( 1.42 \times 10^{-13} \) ergs for glass-water-polystyrene contact (Sharma et al. 1992), where 1 erg = \( 10^{-7} \) N·m. Because the flow cell is made of polypropylene, \( A \) is estimated to be \( 1.42 \times 10^{-20} \) N·m.

The separation between the test particle and the support domes results from the formation of a liquid film between the surfaces. The thickness of the film depends on
temperature, pH, and electrolyte (Churaev et al. 1987), surface roughness (Matthewson 1988), and particle size (Tokunaga 2011). Matthewson (1988) estimated the thickness of liquid film between a sphere and a flat glass surface to be in the range of 2 to 100 nm. Churaev et al. (1987) reported a range of 10 to 250 nm for quartz. The particle separation distance can also be affected by flow-induced agitation. Figure 4-3 shows the variation of the van der Waals force when $D$ varies from 0 to 200 nm.

![Variation of van der Waals force with particle separation distance](image)

Figure 4-3. Variation of van der Waals force with particle separation distance $D$, where $A = 1.42 \times 10^{-20}$ N·m, $R_f = 0.345$ mm and $R_s = 0.188$ mm.

### 4.2.2 Electrostatic Force

The positive electrostatic force (repulsion) between two spheres is expressed using (Israelachvili 2011):
\[ P_{\text{electrostatic}} = \kappa \left( \frac{R_1 R_2}{R_1 + R_2} \right) Z e^{-\kappa D} \tag{24} \]

Where \( \kappa^{-1} \) = the Debye length of aqueous solution (m)

\( Z \) = the interaction constant (N)

The Debye length is the measure of a charge carrier’s net electrostatic effect in solution and how far those electrostatic effects persist, and it can be expressed by (Israelachvili 2011):

\[ \kappa = \left( \frac{2N_A z^2 e^2 M}{\epsilon_0 \epsilon K T} \right)^{1/2} \tag{25} \]

Where

\( N_A \) = Avogadro’s constant = 6.022140857 \times 10^{23} \text{ mol}^{-1}

\( z \) = the valence of the electrolyte

\( e \) = the charge of an electron = 1.60217662 \times 10^{-19} \text{ coulombs}

\( M \) = concentration of electrolyte (mol/m³)

\( \epsilon_0 \) = 8.854 \times 10^{-12} \text{ (a constant unitless parameter)}

\( \epsilon \) = dielectric constant (permittivity) of a medium at zero frequency

(unitless; see Table 4-1)

\( K \) = Boltzmann’s constant = 1.38064852 \times 10^{-23} \text{ N⋅m/K}

\( T \) = absolute temperature (K)
Table 4-1. Dielectric Constant values of water at 1 atm pressure (Israelachvili 2011)

<table>
<thead>
<tr>
<th>T</th>
<th>0°C (273.15K)</th>
<th>10°C (283.15K)</th>
<th>20°C (293.15K)</th>
<th>25°C (298.15K)</th>
<th>37°C (310.15K)</th>
<th>50°C (323.15K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ε</td>
<td>87.9</td>
<td>84.0</td>
<td>80.2</td>
<td>78.4</td>
<td>74.2</td>
<td>69.9</td>
</tr>
</tbody>
</table>

The interaction constant can be expressed by (Israelachvili 2011):

\[
Z = 64\pi \varepsilon_0 \varepsilon \left(\frac{KT}{e}\right)^2 \tanh^2 \left(\frac{ze\zeta}{4KT}\right)
\]

(26)

Where \( \zeta \) = the zeta potential, which depends on pH, ionic strength, and the material.

In this research, attempts to directly measure particle separation distance and zeta potential were unsuccessful. Therefore, the electrostatic force on a spherical particle is estimated using the parameter values outlined in Table 4-2.
Table 4-2. Parameter Values used in Calculating the Electrostatic Force on a Particle Under the Effects of Particle Separation Distance, pH, Ionic Strength, and Temperature

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Parameter values under the effects of</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Particle separation distance</td>
</tr>
<tr>
<td>$R_1$ (of test particle) (m)</td>
<td></td>
</tr>
<tr>
<td>$R_2$ (for supporting particle) (m)</td>
<td></td>
</tr>
<tr>
<td>$N_A$ (mol^{-1})</td>
<td></td>
</tr>
<tr>
<td>$\varepsilon$ (unitless)</td>
<td></td>
</tr>
<tr>
<td>$\varepsilon_0$ (unitless)</td>
<td></td>
</tr>
<tr>
<td>$\varepsilon$ (unitless)</td>
<td>80.2, based on temperature of 20°C in Table 4-1</td>
</tr>
<tr>
<td>$K$ (N·m/K)</td>
<td></td>
</tr>
<tr>
<td>$T$ (K)</td>
<td>293 is selected, based on the temperature range in</td>
</tr>
<tr>
<td></td>
<td>the experimental program, 282.9 to 323.15 K</td>
</tr>
<tr>
<td>$M$ (mol/m^3)</td>
<td>100 mol/m^3 is selected for ionic strength $I = 0.1$ mol/L, based on the range of $I$ from 0.002 to 0.150 mol/L in the experimental program.</td>
</tr>
<tr>
<td>$\zeta$ (N·m/coulombs)</td>
<td>-37 × 10^{-3} as a benchmark value. For glass in fluids with ionic strength of 0.01 to 0.1 mol/L and pH of 5 to 10, $\zeta$ is between -20 to -37 mV, or (-20 to -37) × 10^{-3} N·m/coulombs (Sharma et al. 1992).</td>
</tr>
<tr>
<td></td>
<td>A constant value of -37 × 10^{-3} N·m/coulombs is selected, due to lack of measurement of $\zeta$ of fluids with different ionic strengths.</td>
</tr>
</tbody>
</table>
4.3 Effects of Particle Separation Distance, pH, Ionic Strength, and Temperature on Electrostatic Forces

4.3.1 Electrostatic Force Under the Effect of Particle Separation Distance

As shown in Equation (24), the electrostatic force between two spheres depends on the separation distance ($D$) between the two spheres. The sensitivity of electrostatic force to $D$ is shown in Figure 4-4. As $D$ increases from 1 to 30 nm, the electrostatic force decreases from $10^{-7}$ to $10^{-20}$ N. The parametric analysis presented in the following sections assumes a value of $D = 2.5$ nm.

Figure 4-4. Variation of electrostatic force with particle separation distance $D$. 
4.3.2 Electrostatic Force Under the Effect of pH

This section investigates the effect of pH on the electrostatic force. Zeta potential ($\zeta$) in Equation (26) depends on a solution’s pH; the dependence is generally illustrated in Figure 4-5. More specifically, Sharma et al. (1992) found that for glass in fluids with ionic strength of 0.01 to 0.1 mol/L and pH of 5 to 10, $\zeta$ is between -20 to -37 mV, or (-20 to -37) $\times 10^{-3}$ N·m/coulombs. In this research, a range of $\zeta$ from -10 to -40 mV was studied. Figure 4-6 shows that for $D = 2.5$ nm, electrostatic force increases by an order of magnitude as zeta potential decreases from -10 to -40 mV. Figure 4-6 also shows that for $D = 2.5$ nm, the negative (attractive) van der Waals force, computed using Equation (23), is unaffected by zeta potential. Furthermore, as zeta potential increases, the net surface force on the test particle can change from attraction to repulsion, as described in Equation (22).

Figure 4-5. Illustration of dependence of zeta potential on pH.
As revealed by Figures 4-5 and 4-6, as pH increases, zeta potential decreases, and the electrostatic force (repulsion) between the test particle and the bed of particles increases. As the repulsion increases, the test particle is more likely to mobilize, i.e., the critical flow velocity decreases. This trend agrees with the experimental observation on the effect of a fluid’s pH on a granular particle’s incipient motion: pH has an inverse relationship with critical velocity, i.e., as pH increases, critical velocity decreases, and particle mobilization is more likely to occur.
4.3.3 Electrostatic Force Under the Effect of Ionic Strength

This section investigates the effect of ionic strength (I) on the electrostatic force. Figure 4-7 shows the variation of electrostatic force as ionic strength increases from 0 to 1 mol/L (at $D = 2.5$ nm). Similar to the analysis presented in Figure 4-6, Figure 4-7 also shows that for $D = 2.5$ nm, the negative (attractive) van der Waals force, computed using Equation (23), is unaffected by ionic strength. As ionic strength increases, the repulsive electrostatic force generally decreases, and the net surface force on the test particle (see Equation (22)) changes from repulsion to attraction. Thus, the test particle becomes less likely to be mobilized, i.e., critical flow velocity increases. It is noted that when ionic strength increases from 0 to 0.014 mol/L, the electrostatic force and the net repulsive force increase, and the critical velocity decreases. This trend was not captured in the experimental program.
In Figure 4-7, a constant zeta potential of \(-37 \times 10^{-3}\) N·m/coulombs (or -37 mV) is used. To reveal the variation in electrostatic force with changing zeta potential, a lower and an upper bound of zeta potential of -40 mV and -10 mV, respectively, were used to compute the electrostatic force, as shown in Figure 4-8. The trend of electrostatic force variation with ionic strength remains the same. As zeta potential decreases from -10 mV to -40 mV, the electrostatic force increases by one order of magnitude and becomes more significant when compared with the van der Waals force. Since zeta potential decreases when pH increases (as shown in Figure 4-5), Figure 4-8 also reveals that as pH increases, the electrostatic force increases, and the ionic strength has a greater effect on the mobilization of particles.
4.3.4 Electrostatic Force Under the Effect of Temperature

Figure 4-9 shows the variation of electrostatic force with zeta potential at two temperatures: 0°C and 50°C. Figure 4-10 shows the variation of electrostatic force with ionic strength at two temperatures: 0°C and 50°C. Both figures reveal that temperature does not have a significant effect on electrostatic force.
Figure 4-9. Variation of electrostatic force between the test particle and a bed particle with zeta potential ($\zeta$) of the fluid at two temperatures (the forces are shown as absolute values.)
Figure 4-10. Variation of electrostatic force between the test particle and a bed particle with ionic strength at two temperatures (the forces are shown as absolute values.)
Chapter 5

Closing Remarks

5.1 Summary and Conclusions

This dissertation first presented the evolutionary development of an experimental methodology for studying the relative and interactive effects of three physicochemical fluid characteristics (viscosity, pH, and ionic strength) on the incipient motion of granular particles under laminar flow conditions. Critical flow velocity was used to quantify the relative erosive capacity of the test fluids. To identify the potential relative and interactive effects, a major goal throughout the research was to decrease the variability in measuring critical velocity of the test fluids. This goal guided four phases of evolutionary developments in the methodological and analytical design and implementation. A robust statistical design of experiments, the response surface methodology (RSM), was used to design and implement 20 test fluids of various combinations of the three experimental factors (viscosity, pH, and ionic strength), whose targeted values were highly controlled throughout the experimental program. Incipient motion of a highly spherical glass bead positioned atop a specially designed and manufactured support pocket within a flow cell was observed using microscope video camera. The flow velocity at the particle’s incipient motion was quantified for seven repeated trials for each of the 20 test fluids. A series of regression analyses were conducted on the results to generate a statistical model to describe the relative effects of the three factors and their interactions. An analytical
study was performed to provide a theoretical explanation for the experimental observations. The knowledge gained from this research is summarized as follows:

1. The critical flow velocity of a granular particle’s incipient motion under laminar flow conditions is highly sensitive to the particle shape and the bed condition on which the particle rests. High sphericity of the test particle and a precisely controlled particle bed aided in realizing a low variation in the observed critical velocity.

2. The response surface methodology proved to be an effective and efficient method to quantify the relative main and interactive effects of the three physicochemical fluid characteristics.

3. Viscosity, pH, and their two-way interaction were determined to be the most influencing factors on the critical velocity. The ionic strength was found to have little impact on the critical velocity.

4. Viscosity and pH were both shown to have an inverse relationship with critical velocity. This is in agreement with the findings of previous studies.

5. Viscosity was shown to be of greater influence on critical velocity than pH.

6. The research revealed an interactive effect between viscosity and pH on the critical velocity. When pH is higher (at 10.5) the viscosity had considerably higher influence on the critical velocity than when the pH was lower (at 3.5).

7. At a viscosity of approximately 0.007 g/cm·s, critical velocity remained relatively unchanged as pH increased from 3.5 to 10.5. At a viscosity greater than 0.007 g/cm·s, critical velocity decreased with an increase in pH. At a viscosity less than 0.007 g/cm·s, the effect of pH on critical velocity reversed;
critical velocity increased with an increase in pH. At pH values from 3.5 to 10.5, critical velocity always decreased with an increase in viscosity.

8. In the test setup employed in this research, the lowest critical velocity occurred at a viscosity of 0.013 g/cm·s (the highest level of viscosity used in all the fluids) and a pH of 7.88 (an intermediate pH value used in all the fluids); the highest critical velocity occurred at a viscosity of 0.00544 g/cm·s (the lowest level viscosity used in all the fluids) and a pH of 4.77 (a medium-low pH value used in all the fluids).

9. Fluids with high viscosity, low pH, and low ionic strength tended to result in the lowest variation in critical velocity, while fluids with low viscosity, low pH, and high ionic strength tended to result in the highest variation in critical velocity.

10. Both electrostatic and van der Waals forces are strongly dependent on the separation distance between two particles. For example, for two particles of $3.45 \times 10^{-4}$ m and $1.88 \times 10^{-4}$ m radius, respectively, the electrostatic force decreases from $10^{-7}$ to $10^{-20}$ N as the distance between the two particles increases from 1 to 30 nm, and the van der Waals force spans between $10^{-7}$ to $10^{-11}$ N when the distance varies from 0 to 200 nm.

11. As pH increases and zeta potential decreases, the electrostatic force (repulsion) between a test particle and a bed of particles increases. As the repulsion increases, the test particle is more likely to be detached and consequently mobilize, i.e., the critical flow velocity decreases.
12. As ionic strength increases, the repulsive electrostatic force generally decreases and could cause the net surface force on the test particle to change from repulsion to attraction. Thus, the test particle becomes less likely to be detached and therefore mobilize, i.e., critical flow velocity increases.

13. The trend of electrostatic force variation with ionic strength remains the same when the fluid’s zeta potential changes from -10 mV to -40 mV. As zeta potential decreases from -10 mV to -40 mV, the electrostatic force increases by one order of magnitude and becomes more significant when compared with the van der Waals force.

14. As pH increases, the electrostatic force increases, and the ionic strength has a greater effect on the mobilization of particles.

15. Changes in fluid temperature between 0°C and 50°C do not significantly affect the electrostatic force with various ionic strength and zeta potential.

5.2 Recommendations

Stemming from the identified/suspected major sources of variation that persisted in the final configuration of the test setup and methodology (see section 3.7), two general recommendations for the continuation of this research are offered:

1. The orientation of the support domes and the test particle could be modified to create a more repeatable resting position from which the particle becomes mobilized. This modification could take the form of a redesign/resizing of the support domes, or their replacement altogether with some alternative support
structure. The perceived challenge would be in the manufacture of the part as the dimensions are small enough to require special machinery and the material of construction must be tough enough to withstand the rigors of the laboratory environment.

2. The manually-operated needle valve could be replaced with some form of automated valve or flow control device that can smoothly and continuously vary the flow rate without imparting any undesired flow perturbations.
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- Peer-reviewed Publications: