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AN EXPLORATION INTO THE CONTROLS AND EXTENT
OF CAPILLARY RISE IN FINE-GRAINED SAND

by

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AN EXPLORATION INTO THE CONTROLS AND EXTENT
OF CAPILLARY RISE IN FINE-GRAINED SAND

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Western Michigan University, 2019

Capillary rise in fine-grained sands is especially important due to the wide distribution of fine-grained sediment throughout the unconsolidated sedimentary layers blanketing the earth. The height to which water rises above the water table in porous media is known as the capillary fringe. Tension pulling on the water molecules from the solid surfaces of pores will cause water to rise in the unconsolidated fine-grained sands until the water reaches equilibrium with the downward force of gravity.

Researching the controls behind capillary rise and being able to predict the extent to which water will rise in fine-grained sand helped provide insight into many applied situations affecting industry, infrastructure, agriculture, and environmental remediation. A review of the literature as well as a series of laboratory experiments helped answer questions surrounding the controls on the extent of capillary rise in fine-grained sands.

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CHAPTER 1

INTRODUCTION

Literature Review and Background Information

Capillary rise in fine-grained sands is especially important due to the wide distribution of fine sands throughout the unconsolidated sedimentary layers covering the earth. The height to which water rises above the water table in porous media is known as the capillary fringe. Tension pulling on the water molecules from the solid surfaces of pores will cause water to rise in the unconsolidated fined grained sands until the water reaches equilibrium with the downward force of gravity.

Researching the controls behind capillary rise and being able to predict the extent to which water will rise in fine-grained sand will help provide insight into many applied situations affecting industry, infrastructure, agriculture, and environmental remediation. A review of literature as well as a series of laboratory experiments will provide insight into the questions surrounding the extent of capillary rise in fine-grained sands.

Significance of Capillary Rise

Evaluating the amount of liquid that will rise in a particular porous medium is important for many reasons. Environmental remediation companies need to know the height of the capillary fringe. The extent of the capillary rise of water above the water table has serious impacts on infrastructure as water and other liquids can degrade foundations. Water is essential to the

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growth of plants and agriculture. Understanding the controls on the movement of water near the water table is paramount.

Water table fluctuations affect microbial processes in soils. The distribution of water and air in the capillary fringe zone has direct effects on the capacity for microbial processes. Microbial activity is directly related to water availability (Sinke et al., 1998). Environmental remediation companies have a strong focus on the capillary fringe zone due to smearing of non-aqueous phase liquids (NAPLs) when the level of the water table fluctuates in soils. This leads to important chemical reactions and biodegradation processes. Capillary rise must be evaluated in site remediation so that any fluids involved in the fluid column (water or contaminants) may be targeted (Adamski et al., 2005).

Adamski et al. (2005) made extraordinary claims that LNAPL accumulations of over 20 ft could be due to gaps or holes in capillary rise of water in silts and clays, holes due to macro-pores or fissures, where the holes in the water-filled capillary fringe would fill up with LNAPL that was perched on top of the fringe. Hence, they claimed that such thick LNAPL accumulations did not necessarily indicate a large LNAPL problem. Dr. Hampton found this article written by consultants and oil-industry employees self-serving, and was eager to critically examine their claims, which were based in part on large values of capillary rise in silts and clays reported in Fetter (1994). Years prior to the Adamski et al. article, Dr. Hampton had called Dr. C. W. Fetter to contest the large capillary rise values he reported for silt and clay. Surprisingly, in 2001 when the fourth edition of Fetter's classic textbook was published, his table of capillary rise values had been removed, and much of the chapter on unsaturated soils had been gutted.

Fluctuating water tables can have devastating effects on foundations of buildings and general infrastructure. A foundation may be built above the water table but could still be within

the capillary fringe. Degradation of the foundation could occur when wetting occurs from the capillary fringe due to the rise of the water table. The soil structure becomes weaker due to the rising water table. The capillary water wets the sediment, and hydrostatic pressure from the in-fringing water leads to the displacement of the foundation sediment (Lockington and Parlange, 2004).

Fluids rising into foundations will also draw into the building materials used to create buildings and other general infrastructure like bridges or dams. Capillary rise can occur in cement, wood, and sediments used to level the base of foundations. Wood will rot, cement will decompose, and foundation sediments will lose structure as fluids infiltrate the materials. Flooring, walls, and insulation are all susceptible to being degraded by water intrusion.

The agricultural sector relies on water to grow crops. Brackish water tables can lead to salts being deposited in soil horizons when the capillary fringe is close to the surface of the earth (Lockington and Parlange, 2004). To properly manage irrigation in regions with brackish water tables, the capillary fringe must be located and capillary forces understood.

Determining Capillary Rise

Figure 1-1 shows a 5-cm diameter glass tube used to see the interaction of water and sand within the cylinder. Figure 1-2 approximates a pore in soils. The extent of capillary rise in a pore is controlled by: the diameter of the capillary tube, the contact angle between the liquid and the wetted surface, density of the liquid, viscosity of the liquid, surface tension, and whether the surface is hydrophobic.

Young-Laplace capillary rise equation.

$$h = \frac{2\gamma \cos\theta}{r\rho g}$$

γ liquid surface tension
ρ liquid density
h liquid height
r capillary radius
g gravity constant
θ contact angle

Low γ decreases contact angle
Low r and ρ increases h   ⇨   Good meniscus requires low γ and r
Alloy has high ρ

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Figure 1-1. Capillary rise test.   Figure 1-2. Young-Laplace equation.

The most important controlling factor of capillary rise is the diameter of the capillary tube. It is an inversely proportional relationship—the smaller the diameter of a capillary tube, the greater the capillary rise. The diameter of the capillary tube in Figure 1-2 is directly related to pore size. Pore size is controlled by grain size distribution and sediment packing.

The wettability of a surface is defined as the angle at which a liquid contacts that surface (i.e., contact angle). The lower the contact angle, the greater the wettability. A contact angle of 0 degrees will allow for a perfectly wetted surface, whereas a contact angle of 180 degrees will produce no wetted surface (Schwartz, 1980). Wetting must occur for capillary rise to take place; capillary rise cannot take place when the wetting angle is 180 degrees, and as the wetting angle decreases toward 0, capillary rise will increase. In most cases the contact angle is assumed to be zero (Hillel, 2007).

Grain Size and Capillary Fringe

Another control of capillary rise is the pore size of a sediment or soil. Larger-grained soils have a more predictable capillary rise, and soils with finer grains are more vulnerable to variability in capillary rise. The rate of capillary rise was greater in coarser-grained soils than finer-grained soils (Salim, 2016). Coarse-grained soils will have higher contact angles than fine-grained soils, and hydrophobic soils will have much higher contact angles than non-hydrophobic soils (Pal and Varade, 1971).

Mathematical Prediction of Capillary Rise

Experiments performed in 1670 by Giovanni Alfonso Borelli showed that the height of water rise in a capillary tube was inversely proportional to the radius of the tube. In 1718, James Jurin confirmed the experiments of Borelli, and formulated an equation that is used to this day to describe capillary rise. This equation which is detailed below is called Jurin's law. Another century elapsed. In 1805, first Thomas Young and then Pierre Simone marquis de Laplace derived an equation describing capillary rise in a more complicated geometry than a cylindrical tube. Their joint equation is called the Young-Laplace equation, but often that title is mistakenly used to refer to Jurin's law, as it was in Figure 1-2 above. Over another century later, the Washburn (1921) equation was derived. It is used to predict the rate of capillary rise. Again, Jurin's law is often mistakenly called the Washburn equation.

All of these equations predict the height or rate of capillary rise in a single capillary tube, which is problematic because soils contain a series of interconnected pore spaces which only can be approximated as capillary tubes.

Hillel (2007) states:

"The height of capillary rise is:

$h_c = (2\gamma \cos \alpha) / (g (\rho_l - \rho_g) \gamma)$ (Eqn. 1-1)

where ρ_g is the density of the gas (neglected)

ρ_l is the density of the wetting liquid

g is the acceleration of gravity

r is the average or representative capillary radius

α is the contact angle

γ is the surface tension

ρ_g is the density of the non-wetting liquid, often a gas like air. Compared to water, the density of

air is essentially zero.

Accordingly, the equation relating the equilibrium height of capillary rise h_c to the average or

representative radius of the pores is Jurin's Law:

$h_c = (2\gamma \cos \alpha) / (r \rho_w g)$　　　(Eqn. 1-2)

where γ is the surface tension

r is the representative capillary radius

ρ_w is the water density

g is the gravitational acceleration

α is the wetting angle, often (though not always justifiably) taken as zero."

A margin of error is involved with these two equations as they are used to estimate rise in

a capillary tube, which is more consistent than measuring capillary rise in soils whose pore

spaces are not as regular. In the laboratory setting it is very difficult to get pore spaces to be

entirely uniform; sediment packing is variable, pore spaces are not similar, and complexities in the

materials comprising the sediment lead to variability in the wetting surfaces within the soil. All these inconsistencies make predicting capillary rise in fine-grained soils very difficult, especially when using mathematical models to predict capillary rise in finer-grained sediments with longer capillary rise times (Salim, 2016).

Potential capillary rise heights were calculated using Hillel's (2007) equilibrium height of capillary rise equation (Eqn. 1-2). The height of capillary rise (Table 1-1) was estimated for different r values ranging from 28-31 micrometers (capillary radius), using γ (the average surface tension of de-ionized water) = 72.2 dynes/cm, α (the wetting angle) assumed to be 0 degrees, ρ_w (the density of de-ionized water) = 0.9982 g/mL, and g (the acceleration of gravity) = 980.7 cm/sec^2.

Table 1-1. Equilibrium Height of Capillary Rise Equation

$h_c = (2\gamma \cos\alpha)/(r\rho_w g)$	(Hillel, 2007)
Height of capillary rise	h_c = 47.62 cm - 50.9 cm
Average surface tension of de-ionized water	γ = 72.2 dynes/cm
Wetting angle, assumed to be 0	$\alpha = 0$
Range of capillary radius values	r = 0.0028 cm - 0.0031 cm
Acceleration of gravity	g = 980.7 cm/s^2

Inputting the range of r values of varying capillary radius into the equilibrium height of capillary rise equation, we get a range of capillary rise heights between 47.62 and 50.9cm. It is expected that under the best conditions, constrained by using sand grains averaging 100 mesh

size, or 0.149mm in diameter, capillary rise heights would be at least 47cm and should not exceed 51cm.

While there are several variables that control the capillary rise height, the focus of this research is on grain size and the resulting pore or capillary radius. Another variable of interest is the type of fluid used. When the type of fluid is held constant, the values for the tension of de-ionized water have a very narrow range, leaving grain size and capillary radius as the changing variable. After running capillary rise experiments with a known grain size, capillary radius can be solved for using the above equation and the measured capillary rise.

Rearranging Eqn. 1-2 to solve for capillary radius:

$r = (2\gamma \cos \alpha) / (h_c \rho_w g)$ 　　　　(Eqn. 1-3)

Using measured values for the height of capillary rise and the surface tension allows the capillary radius to be calculated. This is equivalent with what Fetter (1994) did in Table 1-2 below, except he calculated the height of capillary rise using different radius values. He assumed that the capillary radius value was one-fifth of the average grain size, except for fine silt.

Table 1-2. Fetter Table, 1994

Sediment	Grain Diameter (cm)	Pore Radius (cm)	Capillary Rise (cm)
Fine silt	0.0008	0.0002	750
Coarse silt	0.0025	0.0005	300
Very fine sand	0.0075	0.0015	100
Fine sand	0.015	0.003	50
Medium sand	0.03	0.006	25
Coarse sand	0.05	0.01	15
Very coarse sand	0.2	0.04	4
Fine gravel	0.5	0.1	1.5

As seen in Tables 1-1 and 1-2, a fine sand would be expected to have a capillary pore radius of 0.003 cm (30 µm) and a capillary rise of 50 cm with a grain diameter of 0.015 cm (or 0.15mm).

Defining Fine-Grained Sand

Numerous methods have been used to measure grain size, including: caliper diameter, sieve diameter, weight, volume, equivalent spherical volume and equivalent spherical diameter. Commonly, fine-grained sediments are evaluated by finding the bulk sediment particle size distributions after measuring hundreds or millions of grains by mechanical, optical, or instrumental methods, in which case sediment size is reported in percentage weight or percentage volume (Blott and Pye, 2012).

There is disagreement on a universal method to describe particle size and characteristics of sediments. Most geologists use an adaptation of Wentworth (1922) to describe grain size in congruence with Folk (1954) to describe sediment textures (Blott and Pye, 2012).

Many sediment classification schemes have been developed through the years by a multitude of authors. Their work has resulted in different agencies adopting similar classification techniques. The Bureau of Soils, United States Department of Agriculture (USDA), British Standard for Soil Classification, United States Bureau of Public Roads, American Association of State Highway Officials (AASHO), American Society for Testing and Materials (ASTM), United States Civil Aeronautics Administration (CAA), United States Federal Aviation Agency (US FAA), United States Army Corps of Engineers (USACE), all have had different classification schemes for defining sediment size (Blott and Pye, 2012).

The Soil Science Society of America (SSSA) set up an international committee in 1962 to establish a uniform classification scheme, at the same time the Society of Economic Paleontologists and Mineralogists set up a committee to do the same thing. Representatives from SSSA, SEPM, ASTM, Geological Society of America, Highway Research Board, AASHO, and others came together to organize a unified sediment classification. No collective position was reached, as each organization had too much invested in their own classification schemes (Blott and Pye, 2012).

C. W. Fetter, in his third edition of *Applied Hydrogeology* (1994), quantifies fine-grained sand size a few ways: a grain-size distribution chart based on sieve sizes (Fig. 1-3), particle diameter using the Phi scale (Fig. 1-4), an engineering grain-size classification (Table 1-3), and a definition of sediment size. Figure 1-3 is a grain size distribution chart plotting grain size on a logarithmic scale. Finding the grain-size distribution of fine-grained sand is done by shaking sieves with declining sieve sizes. Figure 1-4 is a classification scheme used by many sedimentologists based on the Phi scale, a logarithmic translation of the Udden-Wentworth classification system (Fetter, 1994).

Table 1-3 displays the definition of sediment grain size based on the American Society of Testing Materials classification scheme. Table 1-2 lists common unconsolidated sediment sizes, and then asserts values for mean grain diameter and pore radius of common sediments, and the expected value of capillary rise based on Equation 2 from earlier in this chapter:

$$h_c = (2\gamma \cos \alpha) / (r \rho_w g) \qquad \text{Equation 1-2}$$

where γ is the surface tension, r is the capillary radius, ρ_w is the water density, g is the gravitational acceleration, and α is the wetting angle, often (though not always justifiably) taken as zero (Fetter, 1994). Using Table 1-2, grain size was defined by Fetter (1994) as fine-grained sand

Figure 1-3. Grain size distribution chart based on sieve size.

Figure 1-4. Particle diameter using the Phi scale.

with a grain size diameter of 0.015cm, which translates to a sieve size of 100 mesh. Sand grains used in the experiments were sieved from 730 grade Covia sand and W-8 grade Nugent sand with 80 to 120 mesh sieve sizes, with the goal of producing an average of 100-mesh-sieve sized sand grains.

Table 1-3. Sediment Grain Size Based on ASTM

Name	Size range (mm)	Example
Boulder	>305	Basketball
Cobbles	76-305	Grapefruit
Coarse gravel	19-76	Lemon
Fine gravel	4.75-19	Pea
Coarse sand	2-4.75	Water softener salt
Medium sand	0.42-2	Table salt
Fine sand	0.075-0.42	Powdered sugar
Fines	<0.075	Talcum powder

Predicting Capillary Rise and Pore Radius With Laboratory Experiments

Both laboratory experiments and mathematical models are ways of estimating the desired value, the actual capillary rise in the "field". Laboratory experiments are generally more accurate than mathematical models at predicting capillary rise (Salim, 2016). Laboratory experiments may be more realistic because they consider parameters found in the field. Experiments considering finer grained sediments with longer capillary rise times are likely more accurate than mathematical models. However, mathematical models have the advantage of being quicker and easier to run than lab or field experiments.

Laboratory experiments are better than field tests at verifying mathematical models due to greater control on the grain size and sediment packing which determine the pore sizes. A controlled closed system of a glass cylinder is carefully packed with specific sized sediment grains and submerged into a tank of water representing a natural water table in the field. Variables within the closed system, like grain size, liquid type, grain materials, and style of sediment packing, can be changed to test the variability in extent of capillary rise.

Pore radius can be calculated as the result of capillary rise. Putting in the results from the capillary rise tests from the glass cylinders into Equation 1-3 will give us the pore radius between the sand grains. Measuring the surface tension of the water used for each experiment with a tensiometer will add another constraint to accurately measure pore radius. By measuring surface tension, capillary rise, grain size, and calculating pore radius, Fetter's findings of capillary rise of fine-grained sand in Table 1-2 can be tested.

Li et al., 2018, proposed a method to determine maximum capillary rise height by using the soil water characteristic curve to elucidate the microscopic pore distribution. The capillary rise is determined by the interconnected small pore spaces within the soils. An average pore radius calculation was created based on soil water characteristic curve experiments and statistical theory. The equation can help determine the distribution of pores as it relates to the degree to which different soils are compacted. For fine-grained soils the average pore radius calculation is reasonably precise. The maximum capillary rise can be calculated using the soil pore radius. The greatest height of capillary rise is controlled mainly by the distribution of larger pore sizes; the smaller pores have a negligible effect on the overall extent of the capillary rise (Li et al., 2018).

Measuring Capillary Rise in the Field

Getting an accurate and precise measurement of capillary rise in the field is very difficult. Capillary rise is difficult to measure in a laboratory setting using a glass cylinder. Salim (2016) tried using dyes to make the water-wet silt easier to distinguish from dry silt, but the dyes changed the capillary rise. Dang-Vu and Hupka (2005) dyed water using potassium permanganate at a concentration of 0.5 g/l. They didn't mention any effects on water surface tension or density, but it seems likely there would be effects. Ultimately, the best laboratory measurement technique is

shining a bright light through the far side of the tube, which highlights the translucent wetted sand and the opaque dry sand giving the height of capillary rise so it can be measured visually. Perhaps some variant of this technique could be used in the field, but that hasn't been success-fully developed yet. There are many environmental interactions that occur in the field that cannot be replicated in a laboratory, further justifying field work to substantiate laboratory experiments.

Summary

The height of the capillary fringe is difficult to predict, especially in fine-grained soils. Water and other fluids must pass through the capillary fringe zone before entering the water table. More work is required to better understand the interaction of fluids within the capillary fringe zone. Equations used to estimate capillary rise make assumptions that could lead to inaccuracies in estimates of capillary rise in the field. Laboratory experiments described below were performed to control the values of all known important variables to further constrain the extent of capillary rise in fine-grained sediments.

CHAPTER 2

LABORATORY RESEARCH

Introduction

Data on the height of the capillary rise in fine-grained sands varies considerably. Several experimental methods were used to help get an accurate and precise measurement of capillary rise in fine-grained sand. Previous experiments were replicated, methods of research were explored, a description of data analysis was provided, and an in-depth analysis of the findings is presented.

Materials

An extensive amount of materials was needed to run thorough experiments and constrain error. Glass tanks and Plexiglas boxes were used to hold water or kerosene around the bases of the glass columns that were submerged for the capillary rise tests. The Plexiglas boxes had the dimensions of 33.5cm long, 20cm wide, and 33cm high. The glass tank dimensions were 35cm long, 15cm wide, and 20cm high.

The three glass columns were created for Salim (2016) by Meints Glass Blowing, Kalamazoo, MI, and used for the capillary rise experiments (see Appendix A). The dimensions of the columns are 122cm in height and 5cm in internal diameter. Several cleansers were used to remove any residue or contaminants from previous experiments.

Sand from Covia Corporation—an industry leader in aggregate and mineral mining—was used in the initial experiments. The sand was donated from the Benton Harbor, Michigan branch of Covia Corporation and was mined from the St. Peter Sandstone formation outside of Wedron, Illinois. The Covia sand (Fig. 2-1) was very pure, nearly all silica. After using the 730 grade Covia sand for several tests, the initial supply of sand was exhausted and there was not enough to complete the tests necessary for the research.

Figure 2-1. Covia sand, mm scale.

Three 9.5-liter buckets of Covia 730 grade sand were delivered to Western Michigan University (see Appendix B). The Covia sand was chosen due to the promising grain-size distribution data describing the industrial uses and commercial availability. The 730 grade Covia sand has been washed, screened, and processed to be the highest-purity silica and finest-grade sand available. As seen in Figure 2-1, it is nearly pure quartz silica, very uniform in grain size, 0.177-0.125mm (having been sieved twice between 80, 100, and 120 mesh sieves), and displays sub-rounded grain shape.

A more local supplier of sand was sought for the remaining experiments. Nugent Sand Company of Muskegon, MI—a leader in supplying industrial-grade sand to the petroleum industry and foundries—donated their W-8 grade sand.

Six 19-liter buckets of Nugent W-8 grade sand were picked up from the Nugent Sand

Company outside of Muskegon, Michigan (see Appendix C). The Nugent sand was mined from

windblown sand deposits found near the coast of Lake Michigan. The Nugent sand was not pure

silica; lithic fragments are present, and organic detritus was also visible before the sand was

sieved. The sand from the Nugent Sand Company is a better representation of what would be

found in the natural environment. The organic detritus was removed by the sieving process,

again being sieved twice. All that remained after sieving were silica and lithic fragments that

were uniform in grain size between 0.250-0.125mm (passing 60 to not passing the 120 mesh)

and were rounded to sub-rounded in shape (Figure 2-2).

Figure 2-2. Nugent sand after sieving, mm scale.

De-ionized water from the Western Michigan University Chemistry Department was used

for all the experiments except for the final one, which used kerosene. De-ionized water should

be free of all contaminants, constraining error in the surface tension and ensuring consistent

water quality. The cylinders were rinsed, cleaned, and all particles were removed using de-

ionized water. Using cleaning solutions rather than just DI water would potentially change the

water surface tension and affect the overall capillary rise.

A surface tensiometer was used to test and record surface tension values for the de-ionized water and kerosene used in the capillary rise experiments. The Kyowa CBVP-Z model tensiometer (see Appendix D) in the Geochemistry laboratory of Rood Hall, was used to test the surface tension of the liquids used in the capillary rise experiments. The Wilhelmy plate method was utilized (see Appendix D) in all tests. A glass receptacle was used to hold the liquids (see Appendix D). The Kyowa CBVP-Z manual was referenced for proper recording methods, calibration methods, and testing methods (see Appendix E).

Sediment was evenly distributed throughout the columns using a funnel with a 45cm length of PVC pipe attached and a 35cm tube extension (see Appendix A). The PVC pipe and extension allowed for the sand to be distributed more evenly throughout the glass columns. The sand was placed into the funnel which directed the sand into the PVC pipe. The funnel and attached PVC pipe was moved in a circular motion within the column to help evenly distribute the sediment. The columns were also tamped to make sure that sediment was packed as evenly as possible. The columns were held suspended in the air and struck with a closed fist after each scoop of sand was poured in, and tamped again after the column was filled with sand.

All images were captured with a Samsung S7 Edge with a 12-megapixel camera. Videos were taken to observe the surface tension breaking in the tensiometer as well as to observe capillary rise within the interstitial spaces in the sediment.

Face masks were worn with the intention to prevent inhalation of fine-grained sand when pouring sediment between buckets, placing sediment in the funnel, and when actively working with the sieving equipment. Silicosis is a real and present danger when working with fine-grained sand; the face masks prevent inhalation of much fine-grained sediment.

Meter sticks were used to record heights of the water or kerosene in the reservoirs as well as to record the height of capillary rise in the glass cylinders. Caps were placed on top of the columns to reduce evaporation but were vented to not change the inner pressure in the columns.

Nitrile gloves were worn to protect skin from cleaning materials when initially cleaning the glass cylinders, glass tanks, and Plexiglas boxes. Nitrile gloves were also worn when transferring water or kerosene into receptacles to protect hands from contamination. While conducting tensiometer experiments, nitrile gloves were worn both as protection from fluids and as a barrier to contamination.

A new 19-liter kerosene can was purchased and filled with 11 liters of freshly-purchased kerosene. The kerosene was clear and devoid of any contaminants. Kerosene provides a more stable fluid to test capillary rise and constrain the calculation of pore size as its surface tension is more stable chemically than water. Kerosene is safer than gasoline; it is less likely to have incidents as it is not as volatile and flammable. Kerosene is also not as pungent as diesel, providing a better working environment while running experiments in the laboratory.

Aluminum foil was used to cover the reservoirs to limit the amount of error involved with evaporation. The aluminum foil also acted as a barrier to the noxious hydrocarbon fumes from the kerosene.

<center>Experimental Methods</center>

The hard rock laboratory in the basement of Rood Hall at Western Michigan University was used for the laboratory experiments. The hard rock lab is a stable environment with constant temperature, suitable for replicating the experiments with minimal environmental change.

A sieving process was used to separate all varying grain sizes outside of the Udden-Wentworth grain size scale for fine-grained sand, 80-120 ASTM No. (U.S. Standard) sieve sizes (see Appendix F). The shaker machine in the basement of Wood Hall was utilized to sieve the sediment into three grain size fractions between the 60-120 ASTM No. (U.S. Standard) sieve sizes. The screens used had mesh sizes of 60, 80, 100, and 120, with a pan below. The sediments caught on the successively finer 80, 100, and 120 screens were deposited into a bucket and mixed thoroughly. Sediments caught on the 60 screen and the pan were discarded.

Two different types of sand were used, donated from Covia and Nugent Sand Companies. The sand grades were documented and detailed in composition and origin (Appendices B and C).

Five-centimeter internal diameter glass columns were systematically packed with fine-grained sand. A metal sand scoop was used to move sediment from the buckets and pour sand into the columns at a constant rate. The sand was poured into a funnel with a PVC pipe attached to uniformly distribute sediment into the glass columns to make sure sediment grains were packed evenly. Tamping of the columns was utilized to make sure sediment grains were evenly packed.

The glass columns were placed in the glass tanks and Plexiglas box, standing upright, with even liquid height between the reservoirs (between 11 and 12cm).

Deionized water from WMU's Chemistry Building was used in the capillary rise experiments, and kerosene was used as a control to confirm the data on capillary rise of the DI water experiments. When the fluid reaches equilibrium with the atmosphere and stops rising, the height at which the fluid stopped in the sediment was measured in the glass tube relative to the height of the fluid in the reservoir, the difference being the extent of capillary rise.

The surface tension of the liquid in each capillary rise experiment is a major control of capillary rise. It was imperative to get accurate and precise surface tension values for the de-

ionized water and kerosene used in the capillary rise experiments. To ensure accuracy and precision it was necessary to calibrate the tensiometer machine. Two calibration weights, weighing 200mg each, were placed on either side of the Wilhelmy plate. The balance adjustment knob was used to calibrate the balance sensor, being set to a value of "2048" (see Appendix E). After calibrating the balance sensor, the machine was zeroed out. The calibration weights were hung again on the Wilhelmy plate, weighing 399.9mg, confirming the successful calibration (see Appendix E).

To accurately measure the surface tension of the liquids, the Wilhelmy plate needs to be sterilized and free of any contaminants. Denatured alcohol (see Appendix G) was used to clean the surface of the Wilhelmy plate. The denatured alcohol was also used in the alcohol lamp. Grasping the Wilhelmy plate with tweezers, the plate was submerged in a laboratory dish containing denatured alcohol, then allowed to air dry. After drying, the Wilhelmy plate was grasped with the tweezers again and held over the lit alcohol lamp. It was held over the flame until the plate glowed orange, roughly 20 seconds. The Wilhelmy plate was then carried to the plate rest hanger (see Appendix E) and left to cool.

After cooling, the Wilhelmy plate was placed on the intermediate hanger attached to the balancing hook (see Appendix E). The stage position was set so that the laboratory dish containing the liquid sample was between 5 and 10mm below the Wilhelmy plate. The first measurement mode was selected (see Appendix E) as it measures low-viscosity liquids like water and kerosene. The stage begins to move up slowly, the plate makes contact with the surface of the liquid, and the stage continues to move up, wetting the plate. The stage then moves down, after the plate is wetted, and begins measuring the surface tension of the liquid. As

the stage moves to the original position, the plate breaks free from the surface of the liquid, giving the final surface tension value.

The top of water-saturated silica sand is hard to discern under ordinary room lighting. A flashlight app on the Samsung S7 held next to the glass column provided light bright enough to visually measure the height of capillary rise. The difference between the translucent wetted sediment and the opaque dry sediment was taken as the boundary of capillary fringe. Markers were used to mark the top of the capillary fringe on the glass cylinder, or the greatest extent of capillary rise. Pictures were taken using a digital camera. The extent of capillary rise was measured from the top of the liquid in the reservoir to the mark of the capillary fringe on the glass column.

Methods for Processing Fine-Grained Sands

Sand was separated by grain size, using U.S. Standard 60, 80, 100, and 120 size sieves (see Appendix F). A scoop was used to distribute the fine sand grains from buckets into the sieves. The sand was shaken twice for six minutes. At the beginning of this process, the top sieve was filled with sand directly from one of two suppliers. After the first round of sieving, only the sand caught in the 80, 100, and 120 sieve sizes was re-sieved. The sand caught in the 60 sieve (above 80), and the sand that passed through the 120 sieve was discarded. The sand caught in the 80, 100 and 120 sieves after the first round of sieving was placed in a bucket, and poured back into the top sieve to be sieved again. After being sieved twice, the sand from the three sieves, 80, 100, and 120, was placed in a bucket with a lid to preserve the integrity of the grain-size distribution.

The sieve size selection of 80-120 was chosen to create an average sieve size of 100, or 0.015cm, reflecting the grain size used in Table 1-2 by Fetter (1994). The fine-grained sands are a collection of the sieve sizes between 60 and 80, 80 and 100, and 100 and 120. The majority of the sieved sand was between 80 and 100, caught in the 100-mesh sieve. The 100-mesh sieve retained about one-half of the sand used in the capillary rise experiments. The second most represented sand fraction was the grain size between 60 and 80, being caught in the 80-mesh sieve. The 80-mesh sieve retained about one-third of the sand used in the capillary rise experiments. The least represented sand fraction was the grain size between 100 and 120-mesh sieves, caught in the 120-mesh sieve. The 120-mesh sieve caught about one-sixth of the sand fraction used in the capillary rise experiments.

After the sand had been sieved twice and the sand fraction between 80 and 120 mesh was collected, it was placed in a 19-liter bucket with a lid on it. The bucket was shaken, rotated, shaken, and rotated to combine each sand fraction together to be as close to a uniform average of 100 mesh or 0.015cm, reflecting the fine-grained sand size in Table 1-2 from Fetter (1994). The different proportions of sand that were caught between the 80, 100 and 120-mesh screens alters the average slightly. The sand fraction used in the capillary rise experiments is skewed toward the coarse side, but the difference from the initial 100-mesh target should be relatively small.

Determining Contact Angle

The contact angle is usually assumed to be 0 (Hillel, 2007). Assuming a contact angle of 0 throughout each experiment could be a source of error. Pictures were taken of the wetted sand in the introductory experiments to try to measure the contact angle for each experiment. The wetting angles were supposed to be determined using Adobe Illustrator to inspect the images and

determine the contact angle from the wetted sand. This proved to be unreliable and not an accurate way to measure the contact angle.

When analyzing the equations, especially Equation 1-2, it is evident that assuming a contact angle of 0 will not be a great source of error. The cosine of 0 degrees is 1; the cosine of 10 degrees is roughly 0.98, essentially equivalent to 1. When calculating the potential capillary rise heights from Equation 1-2: $h_c = (2\gamma \cos \alpha) / (r \rho_w g)$ and keeping everything constant except for the contact angle, changing it from 0 to 10 degrees shows that for all practical purposes assuming a wetting angle of 0 is acceptable.

49.3cm = (2*(72.2dyn/cm***cos(10)**))/(29.5μm*0.9985g/mL*980cm/sec)

50cm = (2*(72.2dyn/cm***cos(0)**))/(29.5μm*0.9985g/mL*980cm/sec)

Effect of Grain Size on Capillary Rise

Capillary rise increases in fine-grained materials; the grain size directly relates to the pore size or capillary radius (Salim, 2016). Capillary rise data becomes inaccurate as grain size gets smaller. Overall, predictive models become less accurate as the amount of time for capillary rise to reach its maximum extent increases. 80 to 120-mesh U.S. Standard sieves were used to ensure grain sizes meet the standard fine grain size per the Fetter table. Equations from the literature should produce results similar to the extent of capillary rise in the experiments. The equations can then be used to predict the capillary rise in other materials. Finally, an experiment to test the ability to predict capillary rise before testing was run to verify the accuracy and precision of predicting capillary rise using grain-size distribution, and contact angle, before measuring it in a laboratory setting.

Timing of Capillary Rise

For the congruence of laboratory work, it is important to know when fluid has reached equilibrium with the atmosphere. Timing the capillary rise experiments will display what is an appropriate amount of time for the maximum extent of capillary rise to be reached (Salim, 2016). Measurements were taken daily and recorded throughout the experiments. The data was collected, and the length of time for equilibrium to be established was recorded.

Kerosene vs. Water

The capillary rise of both kerosene and water were measured in experiments with fine-grained sands. Reservoirs were filled with deionized water or kerosene, respectively. Glass tubes filled with sand passing the U.S. Standard size 60 sieve but retained by the U.S. Standard size 120 sieve were submerged in the reservoirs to test the effects of water or kerosene, and the differing influence of the respective fluid on capillary rise.

Summary of Research

All data from the laboratory experiments was collected and put into tables on Excel (see Appendix H). It was then analyzed and compared to predictive mathematical equations and the capillary rise table from Fetter (1994). Error percentages were calculated between the methods and displayed in tables. An analysis of the accuracy and precision of the laboratory experiments was conducted. The findings were recorded and compared to the table from Fetter (1994).

CHAPTER 3

RESULTS

Results of Surface Tension Tests

Surface tension values were taken of the liquids used throughout the experiments. The first capillary rise tests were with Covia sand. The surface tension values for the de-ionized water were recorded before the tests were run. With ambient temperatures near 22 degrees Celsius in the laboratory, and the de-ionized water being ambient temperature, it would be expected to see surface tension values around 72.4 mN/m (see Appendix E).

In the first round of tensiometer tests, an average of 69.6 mN/m was calculated after 5 runs of the tensiometer (Table 3-1). The first run of the tensiometer brought the greatest surface tension value, 71 mN/m, and the values declined until bottoming out at 69.1 mN/m (Table 3-1). The water sample used in the surface tension tests was the same de-ionized water used for the first three Covia sand capillary rise tests.

Table 3-1. Covia Sand Capillary Rise Test Data

	Covia Sand Test 1-3 Surface Tension of Water (mN/m)
Run 1	71.0
Run 2	69.3
Run 3	69.4
Run 4	69.1
Run 5	69.1
Average	69.6

The surface tension results of the de-ionized water used in the Nugent sand tensiometer tests differed slightly from the results of the de-ionized water used in the Covia sand tensiometer tests (Table 3-2). The values of the surface tension for the Nugent water tests were slightly higher, between 1 and 1.5 mN/m greater than the surface tension of the Covia water tests. Tensiometer tests were taken of the water used in the Nugent sand-filled glass columns before the columns were inserted into the reservoirs for tests 10, 11, and 12 (Table 3-2). Tensiometer tests were run after the Nugent sand capillary rise experiments 10, 11, and 12 to constrain error and compare the effect the Nugent sand might have had on the surface tension of the de-ionized water in the reservoir, which was the source of the water used in the tensiometer measurements (Table 3-3).

Table 3-2. Water Surface Tension Tests Before Introduction of Sand-Filled Glass Tubes

Nugent Sand Tensiometer Tests	Capillary Rise Test No.		
Surface Tension of Water Before (mN/m)	Test 10	Test 11	Test 12
Run 1	72.0	72.3	72.0
Run 2	70.3	70.1	70.8
Run 3	70.1	69.9	70.7
Run 4	70.2	70.0	70.7
Run 5	70.4	70.4	70.7
Average	70.6	70.5	71.0

The values for the surface tension of the water after being used in the Nugent sand capillary rise experiments (Table 3-3) were slightly higher than the initial surface tension values. On average the surface tension of the water was 0.4 mN/m more after having the sand-filled glass columns introduced to the reservoir. The greatest difference was in test 11, with 0.6 mN/m variance, and the least in test 12 with a variance of 0.2 mN/m.

Table 3-3. Water Surface Tension Tests After Capillary Rise Experiments

Nugent Sand Tensiometer Tests	Capillary Rise Test No.		
Surface Tension of Water After (mN/m)	Test 10	Test 11	Test 12
Run 1	72.1	72.3	72.4
Run 2	70.7	70.8	70.9
Run 3	70.7	70.7	70.9
Run 4	70.9	70.7	70.9
Run 5	70.8	70.8	70.9
Average	71.0	71.1	71.2

Kerosene was used as the control for the capillary rise tests as well as the surface tension tests. Kerosene has a lower surface tension than water, one that is not as susceptible to being affected by contaminants. Very little variance in surface tension values for kerosene was expected, and was witnessed in the tensiometer measurements shown in Table 3-4 below.

Table 3-4. Kerosene Surface Tension Tests Before and After Capillary Rise Tests

	Nugent Sand & Kerosene Capillary Rise Test
	Surface Tension Before Test (mN/m)
Run 1	26.4
Run 2	25.8
Run 3	25.9
Run 4	25.9
Run 5	25.8
Average	26.0
	Surface Tension After Test (mN/M)
Run 1	26.4
Run 2	25.9
Run 3	25.8
Run 4	25.9
Run 5	26.0
Average	26.0

In each of the initial kerosene tensiometer tests, before and after the sand-filled glass column was introduced to the reservoir, the surface tension values were greatest in the first test and diminished after each run of the tensiometer (Table 3-4). There was little variance in the surface tension values of the kerosene between the runs, differing at most by 0.6 mN/m. There was no appreciable difference in the surface tension values before or after the capillary rise tests and the inclusion of the Nugent sand to the kerosene reservoir (Table 3-4).

Results of Capillary Rise Experiments

There were two different sands used in the capillary rise experiments, one from Covia Corporation, and the other from the Nugent Sand Company. The grain-size distributions of the two sands were very similar, but the sand differed compositionally. The Covia Corporation sand was very clean, nearly all quartz, whereas the Nugent Sand Company sand had occasional lithic fragments and biolithic fragments, including visible shell pieces and organic detritus.

The de-ionized water for all experiments was sourced from the supply room in the Chemistry Building on Western Michigan University's campus. The kerosene was freshly purchased from Slim's Stop and Go in Augusta, Michigan and put into a new, clean, portable kerosene can.

There was a limited amount of sand from Covia, only enough to run five capillary rise experiments. The greatest capillary rise reached was 35.7cm and the least was 32.5cm, with an average of 34.6cm (Table 3-5). Each test reached equilibrium in about 72 hours.

The supply of Nugent sand was very generous, about 115 liters, allowing for ample tests to be run. Fourteen tests were run successfully, averaging a capillary rise height of 35.8cm

(Table 3-6). The lowest capillary rise was 32.9cm and the greatest was 41.4cm (Table 3-6). The

standard deviation was 2.45.

Table 3-5. Covia Sand and Water Capillary Rise Measurements

Covia Sand & Water Capillary Rise Experiments	Capillary rise (cm)
Test 1	35.5
Test 2	32.5
Test 3	34.3
Test 4	35.7
Test 5	35.0
Average	34.6

Table 3-6. Nugent Sand and Water Capillary Rise Measurements

Nugent Sand & Water Capillary Rise Experiments	Capillary rise (cm)
Test 1	39.1
Test 2	35.9
Test 3	34.8
Test 4	32.9
Test 5	34.2
Test 6	33.5
Test 7	34.1
Test 8	34.6
Test 9	34.7
Test 10	41.4
Test 11	39.3
Test 12	34.9
Test 13	36.2
Test 14	35.6
Average	35.8

The kerosene control test was run once. Using the clean, fresh, kerosene and twice-

sieved Nugent sand, along with proper sediment packing, the kerosene capillary rise was 19.7cm

(Table 3-7). Great diligence and precautions were taken to reduce any potential contamination and constrain error, to ensure that the capillary rise would reach the greatest height possible.

Table 3-7. Nugent Sand and Kerosene Capillary Rise Measurement

Nugent Sand & Kerosene Capillary Rise Experiment	Capillary rise (cm)
Test 1	19.7

CHAPTER 4

DISCUSSION AND CONCLUSIONS

Comparing Experimental Data With Predictive Models

The data from the experiments was compared with widely accepted capillary rise data, such as Table 1-2 above from Fetter (1994). It was important to use the Jurin's Law equation 1-2 to predict capillary rise before the data from the capillary rise experiments was recorded. It was necessary to have an idea as to what to expect from the capillary rise experiments to know whether the experiments were successful or not. The accuracy of the mathematical model was compared to the experimental data. Comparing Jurin's Law (Eqn. 1-2) with the laboratory experiments helped explain whether the predictive mathematical models could precisely and accurately depict capillary rise.

Table 4-1 gives capillary rise predicted for an assumed small range of capillary radius values for any fine sand, which corresponds to slight variations in grain size or sediment packing in the glass cylinders. This is the same as Table 1-1. Table 4-2 is a numerical analysis of the capillary rise tests for the Covia Corporation sand solving for capillary radius. Equation 1-3 was used in Table 4-2 to evaluate the capillary radius for several capillary rise tests using the Covia Corporation sand by inputting the average measured surface tension of the de-ionized water used in these tests, an assumed contact angle of 0, and the average of the capillary rise measurements (See Appendix H or Tables 3-1 and 3-5 for full tables of results).

Table 4-1. Range of Expected Capillary Rise For Any Fine Sand

$h_c = (2\gamma \cos \alpha) / (r\, \rho_w\, g)$	(Hillel, 2007)
Height of capillary rise	$h_c = 47.6 – 52.7$ cm
Average surface tension of de-ionized water	$\gamma = 72.2$ dynes/cm
Wetting angle, assumed to be 0	$\alpha = 0$
Assumed capillary radius range	$r = 0.0028$ cm - 0.0031 cm
Acceleration of gravity	$g = 980.7$ cm/s^2

The results from the cylinder tests and Equation 1-2 can be compared to Table 1-2 above from Fetter (1994) for fine-grained sand. The expected capillary rise for fine sand from Fetter is 50cm; Table 4-2 uses the average experimental capillary rise of 34.6cm. The pore radius shown in Fetter is 0.0030cm; the average pore radius for these experiments with slightly coarser sand is calculated to be 0.0041cm.

Table 4-2. Capillary Radius Value of Covia Sand Tests Based on Experimental Data

Covia Sand Tests $r = (2\gamma \cos \alpha) / (h_c\, \rho_w\, g)$ Eqn. 1-3		
ρ_w = Fluid Density		γ = Surface Tension
α = Contact Angle		h_c = Capillary Rise
r = Capillary Radius		g = Gravitational Constant
Tensiometer Readings (mN/m)	Average	69.6
Capillary Rise (cm)	Average	34.6
Pore Radius (cm) of Tests 1–5	Calculated	$r = 0.0041$

The capillary rise experiments were run to test how high water could rise due to capillary action in the most ideal laboratory conditions to compare with Table 1-2 above from Fetter's 3rd Edition (1994). Fetter states that a grain size of 0.015cm diameter should have a pore throat of 0.003cm and a capillary rise of 50cm. In Table 4-2, the Covia sand pore throat was calculated to be 0.0041cm, slightly more than the pore throat for fine sand from Fetter. The surface tension readings taken of the de-ionized water in the tensiometer tests averaged 69.6 mN/m; this value was also used in Table 4-3 as input into Equation 1-2. In Table 4-3, the average capillary rise for the Covia Sand tests was calculated using Jurin's Law (Eqn. 1-2).

Table 4-3. Expected Capillary Rise in Covia Sand Based on Experimental Data

Covia Sand Tests	
$h_c = (2\gamma \cos \alpha) / (r \, \rho_w \, g)$ (Eqn. 1-2)	(Hillel, 2007)
Predicted capillary rise	$h_c = 34.7$ cm
Average surface tension of de-ionized water	$\gamma = 69.6$ dynes/cm
Wetting angle, assumed to be 0	$\alpha = 0$
Capillary radius value (Table 4-2)	$r = 0.0041$ cm
Density of de-ionized water	$\rho_w = 0.9982$ g/mL
Acceleration of gravity	$g = 980.7$ m/s^2

The average measured capillary rise for the Covia sand tests was 34.6cm; the results from Equation 1-2 in Table 4-3 show an expected capillary rise of 34.7cm. The intra-granular capillary radius of the Covia sand is the over-riding control of the capillary rise in the experiments. The capillary rise in the Covia sands did not reach the height that was expected by Fetter, due to the

grain size diameter being greater than 0.015cm and the intra-granular capillary radius being greater than 0.003cm. The data from the mathematical model and capillary rise experiments show that Jurin's Law (Eqn. 1-2) for capillary rise predicts the capillary rise for the Covia sand tests very well.

In total there were 14 capillary rise tests done with the Nugent Sand. The average capillary rise value was 35.8cm. The average value of the surface tension for the de-ionized water used in tests 10-14 was 70.9 mN/m. Assigning the average values from the Nugent Sand tests to Equation 1-2, a pore radius of 0.0040cm was calculated (Table 4-4). Similar to the findings from the Covia Sand tests, the Nugent Sand lab tests' capillary rise was lower than would be expected from Fetter, but agrees with the mathematical model as shown in Table 4-5.

Table 4-4. Capillary Radius Value of Nugent Sand Based on Experimental Data

Nugent Sand Tests $r = (2\gamma \cos \alpha) / (h_c \rho_w g)$ Eqn. 1-3		
ρ_w = Fluid Density α = Contact Angle r = Capillary Radius		γ = Surface Tension h_c = Capillary Rise g = Gravitational Constant
Tensiometer Readings (mN/m)	Average	70.9
Capillary Rise (cm)	Average	35.8
Pore Radius (cm) of Test 10-14	Calculated	$r = 0.0040$

The average measured capillary rise for the Nugent Sand tests was 35.8cm. Using the values calculated for capillary radius as well as the average surface tension values, a capillary rise of 36.2cm (Table 4-5) would be expected. This is less than the 50cm expected from Fetter (1994), but is explained well by the capillary radius value being greater than 0.0030cm and the surface tension value being less than 72.2mN/m. As was the case for the Covia Sand, the

Nugent Sand tests reveal that Jurin's Law (Equation 1-2) works well to predict capillary rise in fine-grained sands.

Table 4-5. Expected Capillary Rise in Nugent Sand Based on Experimental Data

Nugent Sand Tests	
$h_c = (2\gamma \cos \alpha) / (r\, \rho_w\, g)$ (Eqn. 1-2)	(Hillel, 2007)
Height of capillary rise	$h_c = 36.2$cm
Average surface tension of de-ionized water	$\gamma = 70.9$ dynes/cm
Wetting angle, assumed to be 0	$\alpha = 0$
Capillary radius value	$r = 0.0040$cm
Density of de-ionized water	$\rho_w = 0.9982$ g/mL
Acceleration of gravity	$g = 980.7$m/s^2

Comparing Kerosene Vs. Water Capillary Rises

Kerosene was used as a control on the water capillary rise experiments because it is more stable than water. Water has a surface tension that is very high, and decreases substantially when water dissolves contaminants, or surfactants. Kerosene's surface tension values are more stable (Table 3-4), allowing for more accurate representation of capillary radius and capillary rise calculations as follows.

Table 4-6 reports the results of all capillary rise experiments done in this work. The capillary rise in the kerosene experiment was lower than in the water experiments primarily because kerosene's surface tension is lower than water's surface tension. The average surface tension values for water were 69.6 dynes/cm (Table 4-3) for the Covia sand tests and 70.9

dynes/cm (Table 4-5) for the Nugent sand tests, whereas the average surface tension for kerosene

was 26 dynes/cm (Table 3-4).

Table 4-6. Comparing Measured Capillary Rises Between Water and Kerosene Tests

Comparison of Water vs. Kerosene Experiments	Average in cm
Covia Sand & Water Capillary Rise Experiments	34.6
Nugent Sand & Water Capillary Rise Experiments	35.8
Nugent Sand & Kerosene Capillary Rise Experiment	19.7

Table 4-7 calculates the capillary radius of Nugent sand based on capillary rise and

surface tension measurements, and assumed values for kerosene density and contact angle. The

capillary radius is smaller than in other experiments, 0.0033cm instead of 0.0040-0.0041cm. This

could be due to errors in the kerosene density, assumed to be 0.81 gm/ml, and contact angle,

assumed to be 0. Other workers have shown that when water is not present, kerosene becomes

the wetting phase, for which a contact angle of 0 would be appropriate.

Table 4-7. Calculated Capillary Radius of Nugent Sand and Kerosene

Nugent Sand Kerosene Test $r = (2\gamma \cos \alpha) / (h_c \rho_w g)$ Eqn. 1-3		
ρ_w = Fluid Density α = Contact Angle r = Capillary Radius		γ = Surface Tension h_c = Capillary Rise g = Gravitational Constant
Tensiometer Readings (mN/m)	Average	26
Capillary Rise (cm)	Average	19.7
Calculated Pore Radius	Calculated	$r = 0.0033$

Table 4-8 shows the predicted capillary rise height for kerosene, 21.8cm. Tables 4-3 and

4-5 show the predicted capillary rise height for water, 34.7cm and 36.2cm, respectively. The

capillary rise heights from the predictive equations for both water and kerosene (Tables 4-3, 4-5, and 4-8) are calculated based on surface tension values recorded in the laboratory (Appendix D) and calculations for capillary radius values (Eqn. 1-3) based on the results of these capillary rise experiments.

Table 4-8. Predicted Capillary Rise of Nugent Sand Based on Experimental Data

Nugent Sand and Kerosene Test Prediction	
$h_c = (2\gamma \cos \alpha) / (r\, \rho_w\, g)$ (Eqn. 1-2)	(Hillel, 2007)
Calculated height of capillary rise	$h_c = 19.8$cm
Average surface tension of kerosene	$\gamma = 26$ dynes/cm
Wetting angle, assumed to be 0	$\alpha = 0$
Capillary radius value	$r = 0.0033$cm
Density of kerosene (assumed)	$\rho_w = 0.81$ g/mL
Acceleration of gravity	$g = 980.7$ cm/s^2

There is congruence between the capillary rise heights recorded in the capillary rise experiments (Table 4-6) and the predicted capillary rise heights from the mathematical models (Tables 4-3, 4-5, and 4-8). The predictive models for the water capillary rise experiments were close, only varying by 3%. The kerosene predictive equation varied even less. It is reasonable to say that the equation used to predict capillary rise is substantiated by the results of the water and kerosene capillary rise experiments.

Predicting Capillary Rise Using Experimental Data

The capillary rise of fluids in sediment can be predicted from known capillary rise heights and surface tension values. Using the data collected from the water capillary rise experiments and tensiometer tests, capillary rise for kerosene in a fine sand can be predicted by comparing the density and surface tension of kerosene and water with a known water capillary rise in that fine sand (Table 4-9).

Table 4-9 shows the ratio equation used to estimate the capillary rise in two different liquids. Inputting the experimental data for kerosene and solving for the average capillary rise of water will give an estimate of potential capillary rise in water (Table 4-10).

Table 4-9. Estimation of Capillary Rise Using a Ratio Equation

Ratio of surface tension to estimate capillary rise height	
h_w = average water capillary rise height	ρ_k = density of kerosene
h_k = average kerosene capillary rise height	γ_w = average surface tension of water
ρ_w = density of water	γ_k = average surface tension of kerosene
$(h_k \gamma_w)/(\rho_w)) = (h_w \gamma_k)/\rho_k))$	

Table 4-10. Estimation of Capillary Rise of Water in Fine-Grained Sand

Ratio of surface tension to estimate capillary rise height	
h_w = 43.3 cm	ρ_k = 0.81 g/cm^3
h_k = 19.7 cm	γ_w = 70.5 mN/m
ρ_w = 0.9982 g/cm^3	γ_k = 26 mN/m
$(\rho_k (h_k \gamma_w)/(\rho_w))/\gamma_k) = h_w$	

Inputting capillary rise of kerosene, and other data, and solving for h_w, a value of 43.3cm is found for capillary rise of water. This is about half way between what is predicted for

capillary rise in fine-grained sands by Fetter, 50cm, and the average of what was found in the capillary rise experiments, 35.8cm.

Factors Affecting Values From Experiments and Mathematical Models

Capillary rise values of 50 centimeters in fine sands were expected from the literature and the accepted mathematical model, but were not attained in these capillary rise experiments. There are many factors that could affect the capillary rise values, including surface tension, grain size, sediment packing, environmental factors, and sediment composition.

Surface tension of a liquid is a major control on the overall capillary rise of a liquid in sediment. It can be seen in Equation 1-2 that lower surface tension values will lead to lower capillary rise heights. The surface tension of water can be altered by changes in temperature, addition of surfactants, or addition of salts (Bormashenko, 2018). To reduce the amount of variability in surface tension values due to temperature, all these capillary rise experiments were conducted in the hard rock laboratory in the basement of Rood Hall. This room is a stable environment, with very little variability in temperature and no contemporaneous use outside of the capillary rise experiments.

Cleaning agents, which would act as surfactants, were used to clean the testing equipment in the initial experiments. The cleaning agents were removed from the experimental process because they were found to be unnecessary and were a potential source of error. The addition of surfactants into the de-ionized water would reduce the surface tension and reduce the potential capillary rise.

The sediment used in the capillary rise experiments could contain salts and/or soluble metals. The Covia sand was very clean, almost pure quartz, with few lithic fragments visible. The Covia sand came from a sand mine in Illinois where it was mined from a sandstone, then washed, screened, and classified. The product description (Appendix B) displays typical physical and chemical properties of the mined sand, and states the amount of contaminants is negligible, less than 1%.

The Nugent sand was lithic rich and had organic detritus visible on initial inspection. The Nugent sand product description (Appendix C) includes a chemical analysis displaying potential salts and clays that would influence the surface tension of the de-ionized water used in the capillary rise experiments. An increase in dissolved solids, like metals or salts, would increase the surface tension values and ultimately the height of the capillary rise (Bormashenko, 2018). Up to 5% of the Nugent sediment in the capillary rise tests could be salts or soluble metals, including potassium oxide. Surface tension measurements of water were taken before and after capillary rise tests 10-14 to observe the potential effects of contaminants in the Nugent sand (Table 4-11). The greatest difference was found in Run 2 of Test 11 and Run 1 of Test 12, 0.7mN/m and 0.4mN/m, respectively. Although there was an increase in the surface tension, on average it was less than 1%, and was deemed negligible.

Grain size is the primary constraint on capillary rise. When it comes to fractions of sands, the finer the grain size, the greater the capillary rise. To constrain error on grain size, both Covia and Nugent sands were sieved twice and screened to try to create an average of 100 mesh, or 0.015cm grain diameter. Equation 1-3 helps analyze the control on grain size by calculating the pore radius. Table 4-12 shows the distribution of capillary rise heights in relation to capillary radius for each type of sand used in the capillary rise experiments and the table from Fetter. The

Table 4-11. Tensiometer Tests of Water Before and After Nugent Sand Capillary Rise Tests

Nugent Sand & Water Surface Tension Measurements					
Surface Tension Before Test (mN/m)	Test 10	Test 11	Test 12	Test 13	Test 14
Run 1	72.0	72.3	72.0	72.0	72.0
Run 2	70.3	70.1	70.8	70.8	70.8
Run 3	70.1	69.9	70.7	70.7	70.7
Run 4	70.2	70.0	70.7	70.7	70.7
Run 5	70.4	70.4	70.7	70.7	70.7
Average	70.6	70.5	71.0	71.0	71.0
Surface Tension After Test (mN/m)					
Run 1	72.1	72.3	72.4	72.4	72.4
Run 2	70.7	70.8	70.9	70.9	70.9
Run 3	70.7	70.7	70.9	70.9	70.9
Run 4	70.9	70.7	70.9	70.9	70.9
Run 5	70.8	70.8	70.9	70.9	70.9
Average	71.0	71.1	71.2	71.2	71.2

greater capillary radius in the capillary rise experiments is evidence of a grain size greater than the radius corresponding to 0.015cm grain diameter, or a 100-mesh sieve. By charting the Fetter table, and analyzing between the grain size and capillary radius, the approximate grain size can be found and defined for 0.004cm. The discrepancy in capillary rise heights between the tests and Fetter's text could mostly be attributed to the difference in grain size. When analyzing the difference between capillary radius in Table 4-12 there is a 25% difference in capillary radius values between the experiments and the information from Fetter. There is a corresponding difference in capillary rise heights of about 28.8%, showing a nearly linear relationship between the capillary rise heights from the experiments and the capillary rise predicted by Fetter.

Table 4-12. Average Values of Experimental Data Compared to Fetter, 1994, Fine Sand Values

	Nugent Sand	Covia Sand	Fetter, 1994 (Fine Sand)
Avg. Capillary Rise (cm)	35.8	34.6	50
Avg. Capillary Radius (cm)	0.0040	0.0041	0.0030

In Table 1-2, Fetter assumed that the sand grain diameter was five times larger than the pore radius. Taking the average capillary radius found in Table 4-12, 0.004 cm, and multiplying by 5, we get a grain diameter of 0.020cm for the capillary rise experiments. This average grain size of the twice-sieved sand situates it between being a fine sand and medium sand. The difference in grain size explains the difference in pore radius, 0.0040cm calculated from the results of the capillary rise experiments (Table 4-12), instead of 0.0030 from Table 1-2. The capillary rise was ultimately lower in the experiments than in the Fetter table, and is evident in the difference between grain size and pore radius.

The ratio of grain size from what has been calculated from the capillary rise experiments, 0.02cm, to Table 1-2 for fine-grained sand, 0.015cm, is 1.33. When multiplying the ratio of grain size to capillary rise height, (1.33)*34.6cm, a nearly identical number to what should be expected for capillary rise height is found at 46.1cm. This value varies from Fetter's expected capillary rise height for fine-grained sand by 7.7%, or 3.9cm. The relationship between grain size and capillary rise height is nearly linear between Fetter's findings and the results from the capillary rise experiments.

Even with the coarser sand used in the capillary rise experiments, 0.02cm diameter instead of 0.015cm as in Fetter, the relationship between the results in the experiments can be almost directly related to Fetter's findings. Equation 1-2 from Hillel (2007), Jurin's Law, can be used to estimate capillary rise in laboratory experiments. The values in the Fetter table are

nearly harmonious with the capillary rise and pore radius values found with the equations used to estimate pore radius and capillary rise. The initial hypothesis that drove these experiments was that the Fetter table reported capillary rise values that were progressively too high for the finer-textured porous media. The tests show that the values in the Fetter table align with Jurin's Law and what should be expected both in laboratory experiments and most likely in real-world settings.

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Appendix A

Glass Cylinder With Funnel and Tubes to Ensure Proper Sediment Packing

Column-packing funnel with a 45cm length of PVC pipe attached and a 35cm tube extension

Top left: glass column of fine sand recently submerged in water tank. Middle: Two columns of fine sand. Top right: glass column of fine sand previously submerged in water tank showing capillary fringe. Bottom right: close-up of capillary fringe. Bottom left: close-up of capillary fringe.

Appendix B

Covia Sand Information

COVIA

Certificate of Analysis

Analysis:	444450
Grade:	730
Stage:	Shipment
Test Time:	08/26/2018
Customer:	CUSTOMER TEST

Sieve

Sieve (Conforms to ASTME11)	% Retained	Specs	% Retained
20 -USA Std	0.0	0.0 - 0.0	0.0
30 -USA Std	0.0	0.0 - 0.0	0.0
40 -USA Std	0.0	0.0 - 0.5	0.0
50 -USA Std	2.2	0.0 - 6.5	2.2
70 -USA Std	24.3		24.2
100 -USA Std	37.7		37.7
140 -USA Std	24.2		24.2
200 -USA Std	10.2	3.0 - 12.0	10.2
270 -USA Std	1.6	0.0 - 3.5	1.6
PAN -USA Std	0.0	0.0 - 1.0	0.0

Test Details

Test	Value	Spec Min	Spec Max
Grain Fineness	80.95	76.00	85.00

Brandy Harden, Lab Technician Brandy Harden, Operator

Material conforms to specifications. All API RP19C testing qualifies to ISO data and is verified at regular intervals. Refer to current data sheets for specification limits. Direct sales and technical inquiries to:

WEDRON SILICA, J DOLDER QUALITY MANAGER (815)-433-2449 EXT 33389:

WEDRON SILICA CO.

FAIRMOUNT
Minerals

SILICA SAND
WASHED AND DRIED

Typical Analysis - Percent Retained on each Mesh

Microns	Mesh Size	480	460	430	420	410	530	520	510	730
850	20	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
600	30	13.1	0.7	0.1	0.1	0.0	0.0	0.0	0.0	0.0
425	40	73.9	60.7	30.9	18.8	11.2	1.2	0.7	0.0	0.0
300	50	12.0	34.5	45.2	37.5	31.2	25.6	22.2	8.7	2.2
212	70	0.9	3.8	19.4	28.3	33.4	39.9	36.3	27.8	14.7
150	100	0.0	0.3	3.8	12.2	18.2	24.6	27.7	41.7	47.5
106	140	0.0	0.0	0.6	2.9	5.4	7.6	11.2	17.9	28.8
75	200	0.0	0.0	0.0	0.2	0.6	1.1	1.9	3.8	6.4
53	270	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.4
<53	Pan	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
AFS/GFN		30	34	40	46	51	57	60	70	80

Wedron Silica Co. PO Box 119 • Wedron, IL 60557
Telephone: 800-255-7263 • Fax: 269-465-6075
sales@fairmountminerals.com • www.fairmountminerals.com

Typical Physical & Chemical Properties

% LOI	0.07
pH	7.0
ADV	1.0
Grain Shape	Rounded
% SiO_2	99.65
% Al_2O_3	0.065
% Fe_2O_3	0.018
% TiO_2	0.011
% CaO	0.012

Appendix C

Nugent Sand Information

The Nugent Sand Company, Incorporated

4912 Russell Road
Muskegon, MI 49445-9510

(231) 755-1686 Office
(231) 759-0877 Fax

Customer Number:
Customer Name:
Sand Grade: W/802
Date: December 17, 2018
Time: 11:52 AM
Lading Number: TEST SAND

Customer Sand Specifications

Screen	Minimum	Target	Maximum	Screen	% Retained
30	0.00	0.00	1.00	30	0.00
40	0.00	0.10	0.50	40	0.32
50	0.00	2.00	5.00	50	0.81
70	9.00	13.50	19.00	70	9.42
100	40.00	52.00	60.00	100	53.73
140	18.00	27.50	37.00	140	30.52
200	0.00	4.50	9.00	200	4.87
270	0.00	0.50	1.00	270	0.32
PAN	0.00	0.50	0.30	PAN	0.00
A.F.S. Number	77.50	80.00	82.50	A.F.S. No.	80.73
				A.D.V. No.	
				Temperature	

Purchase Order Number:
Test Approved by:
Test Person Initials: GG

Notes: G

The Nugent Sand Company Inc.

Quality & Service Since 1912

Contact
Darrell Brothers, Sales Engineer
Cell: 231-557-4309
Office: 231-755-1209
Email: dbnugsand@aol.com
Mail Address: P.O. Box 1209
Plant Address: 4912 Russell Road
Muskegon, MI 49445

Leader In Industrial Silica Lake Sand Manufacturing

	REPRESENTATIVE PRODUCT BLENDS *(Our blends can be customized to meet client specifications)*														
	390	430	460	480	500	520	550	510	455	W-3	W-4	690	W-5	W-7	W-8
Mesh Size	*PERCENTAGE RETAINED PER SIEVE SIZE*														
30	5.0%	5.0%	3.5%	2.0%	1.5%	1.5%	0.8%	2.0%	2.2%	0.5%	0.4%	1.0%	0.2%	0.2%	0.0%
40	25.0%	19.0%	14.5%	10.0%	6.5%	5.5%	2.2%	10.0%	11.0%	4.5%	1.5%	3.5%	1.5%	1.0%	0.2%
50	50.0%	42.0%	34.5%	31.0%	29.5%	27.0%	24.0%	28.0%	28.0%	16.5%	12.0%	13.0%	7.0%	4.5%	2.0%
70	15.5%	24.0%	33.0%	37.0%	39.0%	41.0%	46.0%	36.0%	30.0%	34.5%	34.0%	24.0%	23.5%	17.5%	13.5%
100	4.0%	8.0%	12.0%	17.0%	20.0%	21.0%	23.1%	19.0%	19.5%	31.5%	36.5%	37.0%	47.0%	50.0%	52.0%
140	0.6%	1.5%	2.0%	2.5%	3.0%	3.5%	3.5%	4.0%	7.5%	11.0%	13.0%	18.0%	18.0%	23.0%	27.5%
200	0.1%	0.3%	0.3%	0.3%	0.3%	0.3%	0.3%	0.8%	1.7%	1.5%	2.0%	3.0%	2.5%	3.5%	4.5%
270	0.0%	0.1%	0.1%	0.1%	0.1%	0.1%	0.1%	0.2%	0.1%	0.2%	0.4%	0.5%	0.3%	0.4%	0.5%
Pan	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.1%	0.1%	0.1%	0.2%	0.1%	0.1%	0.0%
A.F.S. Grain Fineness	39	43	46	48	50	52	55	51	54	60	65	69	70	75	80

(Left axis label: U.S. Sieve Size Analysis)

About Our Company

Since 1912, The Nugent Sand Company has been privately held and known for its high quality customized silica lake sand blends. Our plant in Muskegon, Michigan can produce over 1.5 million tons of sand per year with decades of sand reserves.

website: www.nugsand.com

Typical Chemical Analysis

SiO2	Silicon dioxide	95.08%
Al2O3	Aluminum oxide	2.62%
Fe2O3	Iron oxide	0.19%
TiO2	Titanium dioxide	0.03%
CaO	Calcium oxide	0.18%
MgO	Magnesium oxide	0.08%
Na2O	Sodium oxide	0.41%
K2O	Potassium oxide	1.41%

Logistics & Packaging

Our plant has both truck and rail capabilities. We ship in bulk loads or packaged in 50 lb, 100 lb, 3,000 lb (super sack) bags.

Typical Test Results

ADV:	8 Maximum
Clay:	0.25% Maximum
LOI:	0.35% Maximum
Moisture:	0.20% Maximum
PH:	7-8.5
Fusion Point:	2750 F

Typical Applications

Foundry

3-D Foundry/Additive Manufacturing

Hydraulic Fracturing

Building Products Manufacturing (Roofing, Cement Mortar, Fiberglass)

Geothermal Heating and Cooling

Golf Courses (Top Dressing, Bunker & Divot)

Colored Glass Manufacturing

Sand Blasting

Volleyball Courts / Play Sand / Equestrian

Paint/Coatings Additive

Appendix D

Tensiometer Machine and Wilhelmy Plate

Tensiometer recording surface tension of DI water

Tensiometer Wilhelmy plate in water

Appendix E

Manual for Kyowa CBVP-Z Model Tensiometer

Requirements

- A liquid sample (Use distilled water or pure water for testing)
- Glass laboratory dish
- Detecting plate (Platinum plate), Intermediate hanger
- Forceps
- Alcohol lamp, A lighter to light the lamp
- A Cleaning solvent (Acetone, ethanol, IPA, etc.)
- A wiper
- A waste liquid container

⚠️ **WARNIN**

- Handling of the alcohol lamp and its lighting must be made under supervision or instruction of an expert.   Wrong operation may lead burnt or firing.
- Handling of solvents must be made under supervision or instruction of an expert.   Wrong handling may lead poisoning by inhalation or firing caused by ignition.
- Do not use the cleaning solvent for cleaning the instruments.   The coating or the material of the instruments must be damaged depending on solvent.
- Do not use a fire except that of alcohol lamp for cleaning the platinum plate or the ring.   For example in case of using the gas burner, the plate and the ring must be bent due to its strong fire, and the measuring quality must be inferior.

Alcohol Lamp

lighting	extinguish	supplying alcohol	adjusting wick
Image to bring fire from the lower.	Image to put the cap from the side.	Put up the wick holder. Take care not to spill.	Adjust the wick height with forceps.

Pouring the alcohol for fuel in the alcohol lamp:
- Put up the wick holder made of white ceramic and pour the alcohol about 70% of full amount.   Take care not to spill the alcohol.
- Adjust the height of wick checking the actual fire just lighting.   The wick height about 5 ∼10mm and the fire height about 30∼50mm are proper.

Preparation for Measurement   **BASIC OPERATIONS**

⚠ **WARNING**

- Never use Gasoline   Do not use a fuel except Methyl Alcohol and Ethyl Alcohol for the alcohol lamp.   Using a high volatile fuel like gasoline may lead explosion or fire.

- Care for inflammable matters   Do not use the alcohol lamp near the inflammable matters. (Curtain, papers, solvent, etc.)   Such action may lead ignition and fire.

- Not lay down, not spill   Do not use at the place unstable, or possibly hitting or falling the lamp down.   Setting these place may lead resulting fire.
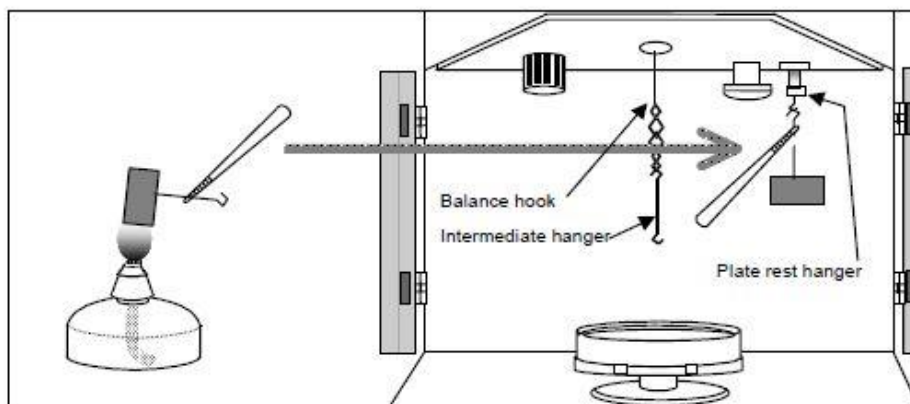
- Never transport while lighting   Do not move the lamp while lighting.   The fuel may be spilled out, which will lead burns and fire.

- Not pull out wick too much   If the wick is pulled out too much, fire must be large, which has possibility of burns and fire.

- Checking fire condition   The fire lighted by alcohol is normally lighted orange color.   When the color is irregular, extinguish at once and check the kinds of fuel.

- Not use for other purposes   Do not use the lamp for other purposes such like heating room or drying materials.   Long lighting may cause lacking oxygen or resulting fire with excessive high temperature.

- Care for storage   Do not keep or use the lamp at the place exposed direct ray of sun or of high temperature.   They must cause quick evaporation of fuel and may lead ignition and fire.

- Correct extinguish   Be sure to check the extinguish after putting the cap on.

Preparation of Liquid Sample

1. Clean up the glass laboratory dish with proper detergent.
   Wipe the inner of the dish by a wipe wetted with solvent.

2. Pour a liquid sample to be measured in the dish and shake slightly imaging to wash the inner of the dish and throw the sample away.   If the sample is available enough, to make this cleaning two or three times is recommendable to remove impurities in the dish.

3. Pour the liquid sample to be measured at least a half of the full amount of dish, then put it on the sample stage of the main unit.

⚠ **CAUTION**

- Take care not to touch the liquid sample, edge or inner of the dish with your fingers. The sample must be contaminated with oily material on the fingers and lead the lower measurement result of surface tension.   Also take care for contaminating especially with oily materials.
- Be sure to pour sample at least a half of the full amount of dish, which is about 7mm. When the volume is not enough, operation errors or irregular resultant may be caused.

Preparation of Detecting Plate (Platinum Plate)

Balance hook
Intermediate hanger
Plate rest hanger

1. Hang the intermediate hanger on the balance hook. (In case of using J-type or H-type constant temp. vessel available at option, the intermediate hanger is not used.)

2. Rinse the platinum plate with solvent and water, then grasp a part of hook of the platinum plate with the forceps and get the plate part into the fire of the alcohol lamp about 30 sec. (By getting the plate into the fire, carbons adhered to the plate surface are burnt out.)

3. After cleaning by fire, the platinum plate must be hung on the plate rest hanger about 60 sec. to radiate and get the plate temperature around ambient.

⚠️ **CAUTION**

- Do not touch the plate with your fingers. The plate must be contaminated with oily material on the finger and lead the lower measurement result of surface tension. Also take care for contaminating especially with oily materials of others.

- Take care not to bend any part of the platinum plate. The surface tension cannot be measured correctly with a bent plate.

- If the measurement should be done at the site prohibiting fire, the cleaning process as below is recommended.:

  Water soluble sample: sufficient rinse with water and cleaning by ultra sonic machine

  Oil soluble sample　　: sufficient rinse and wash with solvent; Acetone, Thinner, etc.

  Alcoholic alkaline solution (Methyl Alcohol of a few percent KOH) is also effective for decomposition of organic matters. Handle the solution under supervision and instructions of an expert.

- When cleaning an inorganic salt group or a sample including metallic elements with the alcohol lamp, remove the material by sufficient washing with water or solvent. Especially, if the plate adhering a Silicon is gotten in fire, SiO_2 must be made on the plate surface, which cannot be removed, and correct surface tension may not be

Adjustment of Sample Stage

Adjust the distance about 5~10mm.

1. After getting the platinum plate cold sufficiently, hang it on the balance hook.
2. Adjust the distance between the plate bottom and the sample surface about 5~10mm. This adjustment is for shortening time of stage movement from starting until contacting the sample with the plate, and does not need precise adjustment.
   Adjustment of the stage is made below.:
   1) Select "01. Measurement" at the main menu.
   2) Press Enter key, then the display shows the message waiting for measurement instruction.
   3) Press Up or Down key depending on the sample stage position. By pressing the keys together with Sub key at the same time, moving the sample stage becomes fast.

⚠️ **CAUTION**

- While operating the sample stage and measurement, do not put the fingers or other things near the stage. Body injury such as wounds may be incurred or damages may be given on the stage section.
- When the plate is happened to contacted with the liquid sample due to excessive raising of the stage erroneously, do steps again from the first at the plate cleaning.
- Be sure to grasp the hook part of the platinum plate with the forceps. Grasping with fingers may contaminate the plate or bend the hook. And grasping the plate part of the platinum plate may cause bending.

Selection of Measurement Mode

```
≪    Main Menu    ≫
02. Measurement Mode [0]
```

1. Select "02. Measurement Mode" at the main menu and press Start/Enter key.   One of the
    Measuring Mode currently selected is displayed as below.

```
≪Measurement Mode≫
01. F.A. :Stab    :Pl (Sta.)
```

2. Ten kinds of modes numbered from 01 to 10 are available as below.   Selection of the mode
    can be made by scrolling with Up Down keys and pressing Start/Enter key.

Mode	Description
01. F.A.:Stab :Pl(Sta.)	⇐ Plate method, Full automatic measure, Standard data out sample of low viscosity like water, alcohol
02. F.A.:Unstab:Pl(Sta.)	⇐ Plate method, Full automatic measure, Continuous data out sample of low viscosity and variable over time like surfactant
03. F.A.:Pull :Pl(Pull)	⇐ Plate method, Full automatic measure, Pull apart method measuring interface tension lower than 5mN/m
04. Auto:Stab :Pl(Sta.)	⇐ Plate method, Automatic measure, Standard data out sample of middle to high viscosity and interface tension
05. Auto:Unstab:Pl(Sta.)	⇐ Plate method, Automatic measure, Continuous data out special solution unable to measure by above 02 mode
06. Auto:Pull :Pl(Pull)	⇐ Plate method, Automatic measure, Pull apart method special sample unable to measure by above 03 mode
07. Auto:Pull :Rg(Pull)	⇐ Ring method, Automatic measure, Pull apart method Platinum ring available at option is needed.
08. Man. :Man :Pl(Sta.)	⇐ Plate method, Manual measure, Manual data out special sample unable to measure by auto or full auto mode like high viscosity liquid
09. Man. :Man :Pl(Pull)	⇐ Plate method, Manual measure, Pull apart method special sample unable to measure by auto or full auto mode like low interface tension
10. Man. :Man :Rg(Pull)	⇐ Ring method, Manual measure, Pull apart method Special sample unable to measure by above 07 mode

➤ Detecting conditions to get the tension:-
   Sta: standard while contacting liquid   **Pull**: pull apart from liquid

➤ Measurement method:-   **Pl**: Wilhelmy plate   **Rg**: du Nouy ring

➤ Data output conditions:-
   Stab: standard equilibrated data   **Unstab**: sequence data with time
   Pull: data just pulling apart   **Man**: manual out

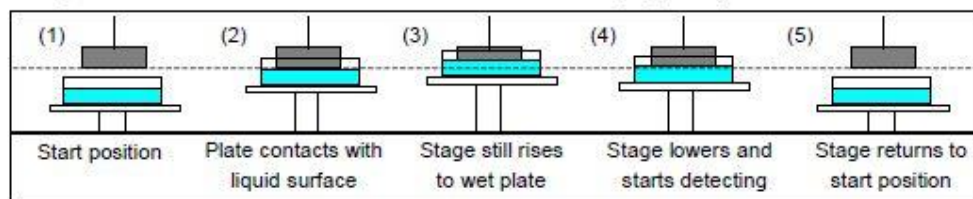➤ Operating condition:-   **F.A.**: full automatic   **Auto**: automatic   **Man**: manual

Operating Conditions

The followings show the movement of the sample stage and the detecting conditions of the measurement modes introduced in the previous page.

Mode 01: F.A.:Stab :Pl(Sta.):

An equilibrated result of surface tension is read out at the step (4) and printed out.

(1)	(2)	(3)	(4)	(5)
Start position	Plate contacts with liquid surface	Stage still rises to wet plate	Stage lowers and starts detecting	Stage returns to start position

Mode 02: F.A.:Unstab :Pl(Sta.):

The stage movement is same as above Mode 01, but reading surface tension is continuously printed out at an interval as per set up.

Mode 03: F.A.:Pull :Pl(Pull):

At the step (4), the stage continues lowering until the plate is apart from the liquid surface, and a peak tension while that action is read out and printed out automatically.

(1)	(2)	(3)	(4)	(5)
Start position	Plate contacts with liquid surface	Stage still rises to wet plate	Stage lowers until plate is apart	Stage returns to start position

Mode 04:Auto :Stab :Pl(Sta.):

An equilibrated result of surface tension is read out at the step (2) and printed out.   To wet the plate with liquid sample compulsory by manual is needed between the steps of (1) and (2).

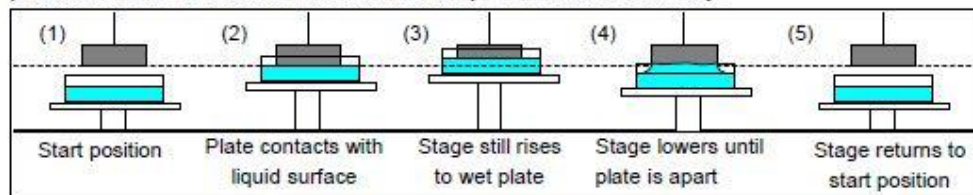| (1)	(2)	(3)	
Start position	Plate contacts with liquid surface and start detecting	Stage returns to start position	Before the step (2), plate is removed and wetted by manual

Mode 05:Auto :Unstab :Pl(Sta.):

The stage movement is same as above Mode 04, but reading surface tension is continuously printed out at an interval as per set up.
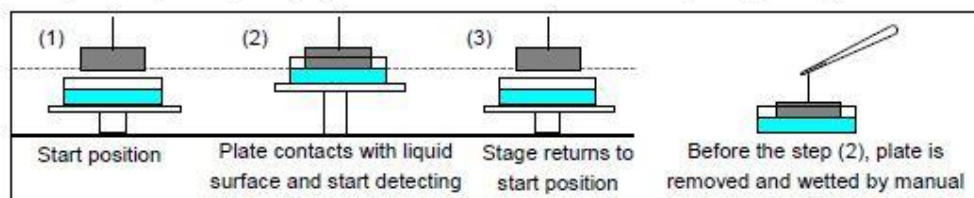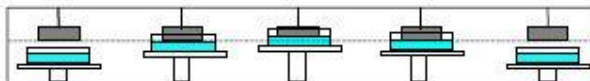
How To Measure 1 –Full Automatic   **BASIC OPERATION** ~~BASIC OPERATION~~

```
≪Measurement Mode≫
01. F.A. :Stab      :Pl (Sta.)
```

Feature & Applications

- This is the superior operation with which the CBVP-Z can get excellent reproducibility. The *Auto wetting* is performed in this mode, which makes the plate wetted with the liquid sample automatically before measuring to avoid measuring errors due to wetting properties.
- Surface tension of low to middle viscosity liquids.

Before Measuring

1. Check that the platinum plate is cleaned and liquid sample is prepared on the sample stage as per described on P21~P24 in this manual.
2. At "02. Measurement Mode" of the main menu, press Start/Enter key.

```
≪Measurement Mode≫
01. F.A. :Stab      :Pl (Sta.)
```

3. Select the 01 mode displayed as in the left figure by scrolling, and press Start/Enter key to determine it.

Start Measurement

```
≪  Main Menu  ≫
01. Measurement      [0]
```

⇩ Start/Enter

```
01. F.A. :Stab      :Pl (Sta.)
Push [Zero] / [Start]
```

⇩ Start/Enter

```
Wait Sensor Stab.
T(s)          1
```

⇩ Automatically

```
Auto Wet:Sense
T(s)          1
```

⇩ Automatically

```
Get PV(Stab)
T(s)          1
```

⇩ Automatically

```
Normal End
Push [Start] / [Main]
```

1. Return to "01. Measurement" of the main menu, then press Start/Enter key for measurement standby.

2. While displaying the message in the left figure, check the preparation of sample and plate again. Then, press Start/Enter key.

3. 0 mN/m is obtained automatically. The message in left figure is being displayed until getting stable of 0 mN/m. The data display will shows "0.0" .

4. The sample stage starts rising, and continues rising after contacting with the liquid surface for *auto wetting*.

5. The stage is brought down a little to get the proper position and measurement is started, then the data display shows the surface tension at real time. The equilibrated surface tension is automatically read out.

6. The sample stage is brought down to the position just starting measurement. The equilibrated surface tension is displayed on the data display and printed out including the date, time and measuring mode.

First of all, try measurement of water surface tension with this 01. Full automatic mode.   Using pure water or distilled water is recommendable.

Surface tension of water is specially delicate to the contamination of oily material, and will be easily lowered with the contamination.   Therefore, to get successful result with measurement of water is the proper training for cleaning the lab. Dish, the platinum plate and also delicate handling of sample.

The following table is the reference list of water surface tension in the literature around the ambient temperature.

Temperature (℃)	20	21	22	23	24	25
Surface tension (mN/m)	72.75	72.60	72.44	72.28	72.12	71.96

MEMO
- The above operations for 01. Full automatic mode is described considering the measurement of water or alcohol.
- In case of measurement such like the following samples, change of the parameters as per described in "Change Parameters" of the chapter "Beyond the Basics" in this manual will be needed.
  - ➢The density of sample is smaller than 0.6 g/cm^3 or larger than 1.4 g/cm^3.
    - → Refer to "Change Parameters (Constant-01)"
  - ➢High viscosity sample (approx. more than 1.5 Pa·s) or surfactant solutions
    - → Refer to "Change Parameters (Constant-02)".

Purpose and Requirement of Calibration

To check the reliability of instrument's accuracy, measurement with the standard matter is needed. And if some difference is occurred, the instrument should be adjusted correctly, which is called *Calibration*. This calibration is to keep the instrument in reliable measurement conditions, and is needed periodically executed.

Generally, water (72.8 mN/m at 20℃) has been used as standard matters for calibrating surface tensiometers. But, as you know, handling the water in good condition to use as standard is difficult because the water surface tension is easily lowered due to easy contamination of itself and also the delicate effects of contamination on the measuring tools. So, this CBVP-Z is used checking of balance loading with the standard weights for calibration procedures.

This chapter "Calibration" explains the calibration procedures for balance unit for detecting surface tension, and also for analog output range of the recorder and vertical moving speed of the sample stage.

Period of Calibration

- When transported the instrument, be sure to make calibration.
- To make calibration at least one time per three months is recommended. And if the instrument is used as frequent as almost every day, to make calibration one time every month is recommended.
- When the instrument is not used for over one month, be sure to make calibration.

Preparation for Calibration

Wait at least 10 minutes after turning on the power. The instrument needs time for warming up to make stable detection. And the following items are required.:

- Detecting parts (Platinum plate or platinum ring)
- Calibration weight (200mg) × 2 pcs. (standardly attached)
  (In case of the ring, 200mg × 4 pcs. are required.)
- Forceps

MEMO
- The calibration menu for the detecting plate and the ring are available respectively. Be sure to make it with the menu to be used. In case of using both the plate and the ring, to make both calibration menu is needed.
- When making calibration of the ring, use the 2 pcs. calibration weights standardly attached to the main unit and also the 2 pcs. attached to the ring at option.
- Do not touch the calibration weights with your fingers, or wet them with liquid. The weights may be corrosion and not keep the correct weight.
- When unpacking the instrument, be sure to make calibration.

Selection of Menu

| ≪   Main Menu   ≫ |
| 05. Calibration   [0] |

⇩   Start/Enter

⇩

| ≪ Calibration ≫ |
| 01. Calib. (A/D)   :Plate |

| ≪ Calibration ≫ |
| 02. Calib. (A/D)   :Ring |

| ≪ Calibration ≫ |
| 03. Calib. (D/A) |

| ≪ Calibration ≫ |
| 04. Calib. (Stage) |

| ≪ Calibration ≫ |
| 05. Calender |

1. Select "05. Calibration" at the main menu, then press Start/Enter key to display the sub menu..

2. The following menus are selected by scrolling with Up or Down key.

Balance calibration for plate method:

Measuring range 0.0 ～ 82.3 mN/m by plate method is calibrated.
※1

Balance calibration for ring method:

Measuring range 0.0 ～ 84.1 mN/m by plate method is calibrated.
※2

Calibration for output range of the analog recorder:

Output voltage of the analog recorder is calibrated in the range from 20mV to 200mV.

Speed calibration for the sample stage movement:

Up/down moving speed of the sample stage is calibrated, with which the parameters related to the stage moving speed are automatically adjusted.

Calendar correction:

Year, month, date and time to be printed with the data are corrected.

※1) Fundamental equation

$P = mg + L\gamma \cdot \cos\theta - h\rho g$

mg = 0 due to zero adjustment and $\cos\theta$ = 1 due to 0° of contact angle.

Therefore, the above equation is explained as

$P = L\gamma - sh\rho g$

Here, h is obtained by spring constant K.

$h = P/K$

$P = L\gamma - s\rho g P/K$

The value of γ upon loading 400mg is calculated out as belows:

$0.4 \times 980.7 = 4.8 \times \gamma - 0.35775 \times 0 \times 980.7 \times 0.4 \times 98037 / 5297$

$$\gamma = \frac{0.4 \times 980.7 + 2.5983}{4.8} = \frac{394.88}{4.8} = 82.27 \leq 82.3 \quad \text{dyne/cm}$$

dyne/cm is explained as mN/m in case of SI unit by MKS.   So, 400mg loading is equal to 82.3 mN/m in case of the CABP-Z.

※2) This is explained in the operation manual for ring method optionally available.

Where:

L: effective plate perimeter 4.8cm

γ : surface tension dyne/cm (mN/m)

s: plate cross section area
2.385×0.015cm^2

ρ : liquid specific gravity (water as 1g/cm^3)

h: plate immersing depth

P: downward forth

K: spring constant 5297 dyne/cm(=mN/m)

g: gravitational constant 980.7cm/s^2

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Calib. (A/D):Plate
W(mg)   0.0 (2048)

Weight   A/D value

Calib. (A/D):Plate
W(mg)   399.9 (2848)

Minimum Value Adjustment (blank for 0 mg)

1. Hang the dried and cleaned platinum plate on the intermediate hanger. (in case of using J-type or H-type constant temp. vessel, the intermediate hanger is not used.)

2. Check the A/D value.:
   This A/D value indicates the position of the balance sensor. "2048" is the fundamental position but 2038~2058 is acceptable.   Adjust the A/D value in the range 2038~2058 by turning the balance adjustment knob.   Turning clockwise gets the value larger.
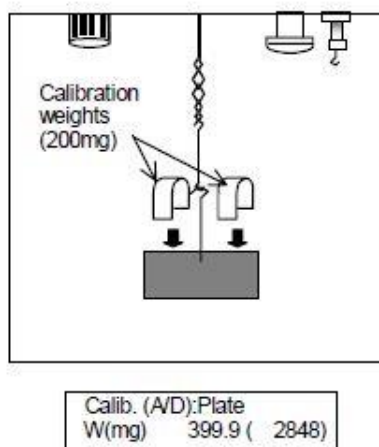   ∗ At this moment, adjustment of weight "0.0" mg is not needed.

3. After confirmed stable of the A/D value, press Zero/Default key, then the weight will show "0.0" mg.
   ∗ Interruption of calibration can be made by pressing Main or Stop key.

Maximum Value Adjustment (span for 400 mg)

1. Put the calibration weights (200mg×2) on the platinum plate as the figure in the left.

2. After confirmed stable of the A/D value, press Start/Enter key, then the weight will show "399.9" mg.
   ∗ At this moment, if the weight shows #399.9", current calibration is correct and pressing Start/Enter key is not needed.

3. Remove the weights and check if the balance loading returns to blank condition (weight shows "0.0" mg) correctly.

4. Press Main key to complete the menu.

MEMO • Tolerance of weight adjustment is within±1.0mg.
   • Calibration of the platinum ring is also made as same steps as the above, but four weights should be put on the ring for calibrating at 800mg, instead of 400mg.

⚠ **CAUTIO**
• Avoid vibration, air flowing and temperature change for correct calibration.
• Be sure to press correct keys for span without fail.   Pressing erroneous key must adjust wrong calibration.

Reference Data   **OTHERS**

Surface tension of water

t(℃)	γ (mN/)	t(℃)	γ (mN/)	t(℃)	γ (mN/)
−5	76.40	19	72.89	40	69.55
0	75.62	20	72.75	50	67.90
5	74.90	21	72.60	60	66.17
10	74.20	22	72.44	70	64.41
15	73.48	23	72.28	80	62.60
16	73.34	24	72.12	90	60.74
17	73.20	25	71.96	100	58.84
18	73.05	30	71.15		

Surface tension of diverse materials

Material	Surrounding phase	t(℃)	γ (mN/m)
Ammonia Dilution (20%)	Air	18	59.3
Ethyl Alcohol	N_2	20	22.27
Olive Oil (d=0.91)	Air	20	32
Glycerin	Air	20	63.4
Chloroform	Air	20	27.28
Acetyl Acid	Air	20	27.7
Diethyl Ether	Its vapor	20	16.96
Carbon Tetrachloride	Air	20	27.63
Dioxane	Its vapor	20	33.55
Petrol	Air	18	26
Toluene	Its vapor	20	22.53
Nitrobenzene	Air	20	43.35
Carbon Disulfide	Air	20	35.3
Paraffin Oil (d = 0.847)	Air	25	26.4
Hexane	Air	20	18.42
Benzene	Air	20	28.86
Methyl Alcohol	N_2	20	22.55
Sulfuric Acid (98.5%)	Air	20	55.1

Reference: 国立天文台編、理科年表　1933、物 29 (449)

Appendix F

Sieving Machine and Covia and Nugent Sands

Covia Sand

Nugent Sand

Pictures displaying similar grain size as seen in the millimeter scale between Covia and Nugent sands, with Nugent sand having more lithic and biolithic fragments.

Appendix G

Solvent Used for Wilhelmy Plate

SUNNYSIDE QUALITY PRODUCTS SINCE 1893

sunnyside

Denatured Alcohol
Alcohol Desnaturalizado

Alcohol Appliance & Marine Stove Fuel

DANGER! ☠ POISON! FLAMMABLE
LIQUID & VAPOR. VAPORS MAY CAUSE FLASH
FIRE. MAY BE FATAL OR CAUSE BLINDNESS IF
SWALLOWED. VAPOR HARMFUL. EYE IRRITANT.
Carefully read and follow all cautions elsewhere
on this container. Use only as directed.

!PELIGRO! ☠ ¡VENENO! LIQUIDO Y VAPOR INFLAMABLES.
LOS VAPORES PUEDEN CAUSAR INCENDIO REPENTINO. PUEDE CAUSAR
MUERTE O CEGUERA EN CASO DE INGESTIÓN. VAPOR NOCIVO.
IRRITANTE DE OJOS.
Lea atentamente y siga las advertencias que se incluyen
en este envase. Use solamente como se indica.

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Appendix H

Tables of Raw Data

	Nugent Sand & Water Capillary Rise Experiments			
	Sand heighth in column (cm)	Beginning reservoir height (cm)	Final reservoir height (cm)	Capillary rise height (cm)
Test 1	73.5	13.0	11.9	39.1
Test 2	73.5	13.2	11.75	35.9
Test 3	73.5	13.4	12.45	34.8
Test 4	x	11.85	10.85	32.9
Test 5	x	11.55	10.7	34.2
Test 6	x	12.5	11.4	33.5
Test 7	x	x	x	34.1
Test 8	x	x	x	34.6
Test 9	x	x	x	34.7
Test 10	x	x	x	41.4
Test 11	x	x	x	39.3
Test 12	x	x	x	34.9
Test 13	x	x	x	36.2
Test 14	x	x	x	35.6
Average				35.8

x = no measurement recorded

	Nugent Sand & Water Surface Tension and Capillary Rise Measurements				
	Test 10	Test 11	Test 12	Test 13	Test 14
Surface Tension Before Test (mN/m)					
Run 1	72.0	72.3	72.0	72.0	72.0
Run 2	70.3	70.1	70.8	70.8	70.8
Run 3	70.1	69.9	70.7	70.7	70.7
Run 4	70.2	70.0	70.7	70.7	70.7
Run 5	70.4	70.4	70.7	70.7	70.7
Average	70.6	70.5	71.0	71.0	71.0
Surface Tension After Test (mN/M)					
Run 1	72.1	72.3	72.4	72.4	72.4
Run 2	70.7	70.8	70.9	70.9	70.9
Run 3	70.7	70.7	70.9	70.9	70.9
Run 4	70.9	70.7	70.9	70.9	70.9
Run 5	70.8	70.8	70.9	70.9	70.9
Average Surface Tension	71.0	71.1	71.2	71.2	71.2
Capillary Rise Height (cm)	41.4	39.3	34.9	36.2	35.6
Average Capillary Rise 10-14 (cm)	37.48				

	Covia Sand & Water Capillary rise height (cm)
Test 1	35.5
Test 2	32.5
Test 3	34.3
Test 4	35.7
Test 5	35.0
Average	34.6

	Covia Sand Test 1-3 & Water Surface Tension (mN/m)
Run 1	71.0
Run 2	69.3
Run 3	69.4
Run 4	69.1
Run 5	69.1
Average	69.6

	Nugent Sand & Kerosene Capillary Rise Test
	Surface Tension and Capillary Rise Measurements
	Surface Tension Before Test (mN/m)
Run 1	26.4
Run 2	25.8
Run 3	25.9
Run 4	25.9
Run 5	25.8
Average	26.0
	Surface Tension After Test (mN/M)
Run 1	26.4
Run 2	25.9
Run 3	25.8
Run 4	25.9
Run 5	26.0
Average	26.0
	Capillary Rise Height (cm)
	19.7