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Effect of particle packing on flow property and strength of concrete mortar

by

Wenjing Cai

A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Civil Engineering (Civil Engineering Materials)

Program of Study Committee: Kejin Wang, Major Professor James E. Alleman Ashley F. Buss

The student author and the program of study committee are solely responsible for the content of this thesis. The Graduate College will ensure this thesis is globally accessible and will not permit alterations after a degree is conferred.

Iowa State University

Ames, Iowa

2017

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ABSTRACT

Optimum particle packing is a key for designing a dense, strong, and durable cement-based material. By optimizing cement and aggregate particle size distribution, the voids among the particles can be significantly minimized, thus increasing packing density, reducing the amount of binder required for filling pores, and improving the material strength, impermeability, and volume stability of the resulting products. Dense particle packing is generally formed by particles with varying particle size distributions, where voids can be successively filled up with smaller particles. Various models have been developed for achieving maximum density, or optimal packing, of aggregate particles in concrete, among which is the Andreasen and Andersen (A&A) model. In concrete practice, groups of aggregate particles with a specific particle size distributions (PSD) are often combined in such a way that the PSD of the blended aggregate is getting as close as possible to a modeled PSD curve.

In this study, modified A&A model is used for achieving optimum packing density of mortars made with various cementitious materials (cement, limestone fines, fly ash, and silica fume) and river sand. The influences of the mortar material proportion on the packing density, flow property, and strength of concrete mortar were investigated. According to the A&A model theory, optimum packing can be achieved when the cumulative PSD obeys equation: P(D) = (Dq-Dq min)/(Dq max-Dq min), where D represents the size of the sieve used for analyzing the particles studied. D min and D max are accounting for the minimum and maximum particle size in the mix, respectively. The distribution modulus q is related to the fineness of the aggregate particles. (Generally, a high q value (>0.50) results in a coarse mixture, whereas a small q value (q<0.25) results in a mixture that is rich in fine particles.

The study consists of two parts, as written of two research papers. In paper one, a fixed distribution modulus (q) value was used, and the PSD of a given mixture was modified by different amount of limestone fines (LFs) and river sand addition. The effects of the LF and sand addition on the particle packing as well as on flow property and strength of concrete mortar were examined. The results indicate that enhanced particle packing improves mortar density and increases viscosity, but it had minimal effect on heat of hydration and yield stress of the mortar mixture. In paper two, various distribution modulus values (q = 0.25, 0.35, and 0.45) were used to evaluate the particle packing quality of a given high performance mortar (HPM). Single sized sand was selected and added to the HPM mixture so as to make the PSD of the modified HPM mixture to the A&A model curve. The minimum sum of squares of the residuals (RSS) was used to assess the quality of the PSD modification. Dry density, rheology, and compressive strength tests were performed for the both original mortar mix and sand-modified mixes. The results show that the increasing q value decreased mortar density, viscosity, and early age strength but had little/no effect on 28-day mortar strength.

CHAPTER 1. INTRODUCTION

1.1 Problem Statement

Since 1892, the statement of that the choice of aggregates influences concrete attracts many researchers to try to find the ideal grading curve. This ideal grading curve should represented the grading with the greatest density. As this scientific approach developed, many packing models are created to generate ideal grading curves. These ideal grading curves are now used for mixture proportion optimization since it is easy to modify the total particle size distribution by adjusting ingredients proportions. However, previous research has shown that the gradation curve which gives the greatest density of the aggregates alone may not necessarily give the greatest density when combined with cement and water due to the way of the cement particles fit into smaller pores in mixture. Therefore, in this thesis, the study of influence of particle packing on flow property and strength of concrete mortar will be investigated, where the mixture particle size distribution is optimized by modifying the gradation of single ingredient without changing ingredients proportion.

1.2 Study Goal and Scope

The thesis addresses following objectives:

- Use modified A&A model to optimize the given samples by changing the gradation of single ingredient with a constant mixing proportion.
- Investigate the influence of particle packing with a fixed distribution modulus on concrete mortar fresh and hardened properties.
- 3. Investigate the influence of particle packing with various values of distribution modulus on concrete mortar fresh and hardened properties.



Following flow chart shows the basic scope of work:

1.3 Outline of Thesis

This thesis is divided into six chapters. Chapter 1 gives a general introduction. Chapter 2 provides a brief literature review of the theory of particle packing, introduction of several packing models, and the effects of particle packing on concrete. Chapter 3 demonstrates the packing analysis for concrete mortar with a fixed distribution modulus, and an investigation of how this particle packing method affect concrete mortar fresh and hardened properties. Chapter 4 presents the packing analysis for concrete mortar with various values of distribution modulus, and a study of the influence of various values of parameter on concrete mortar fresh and hardened properties. Chapter 5 is a summary the study with recommendations for future study.

CHAPTER 2. LITERATURE REVIEW

2.1 Basic Introduction

Nowadays, high performance concrete (HPC) is not only expected to have high strength, but also include early-age characteristics, rheological properties, workability and durability aspects. Due to these characteristics, HPC has been primarily used in construction such as bridges, shotcrete repair, tunnels, tall building and agricultural applications. The constituents of HPC is same as that of normal concrete (such as water, cement, fine aggregate, and coarse aggregate) along with one of the following materials: supplementary cementitious materials, organic admixture, fibers etc. (Mangulkar 2013). Nowadays, more and more attention has been paid to improving the properties of concrete, making it more efficient. Concrete proportioning is a factor in packing problem. With a good mix proportioning, concrete will obtain suitable workability, strength at specified age, maximum density, specified durability and dimensional stability.

Some of the particle packing models, adopted one of several mathematical models to estimate the packing density, provide tools to improve the performance of concrete by reducing free water content and minimize the remaining voids. On the other hand, some of the packing models developed the optimum mixing gradation curves for different mixtures to obtain the highest packing density and the best optimized proportion. It is also possible that particle packing techniques can optimize concrete by reducing the cement content without changing concrete properties in a negative way (Fennis 2006). An optimum packing distribution results in a good packing density with an optimum mixing proportion in an economic way, and also improves workability and reduces the shrinkage and creep.

2.2 Theory of Packing

Packing density (Φ) can be defined as the ratio of the solid volume of the aggregate particles to the bulk volume occupied by the aggregate or as one minus the porosity, which is also in terms of packing (Mangulkar, 2013). In order to increase the particle packing density, the particles with proper sizes should be selected to fill up the voids between the large particles with smaller particles and so forth. Aggregate selection for optimizing packing density could follow empirical tests on various blends of aggregates, mathematical models, or suggested ideal particle size distributions (Koehler 2007). Many of the early researchers, who worked on the particle packing, proposed methods to design and ideal particle size distribution (Fennis 2012). In 1907, Fuller came up with his "Fuller curve"--- an empirical curve for gradation of aggregates, representing the grading with the most optimum density. Fuller curve provides a basic knowledge for further mix design and calculations, and nowadays optimizing concrete mixture to a predefined ideal particle size distribution is still most used in practice and applied in many national standards. However, this optimized gradation will vary along with different types of concrete, rather than be unified for each type of concrete. For instance, the optimum particle size distribution of the mixture with sand from crushed rock and rounded coarse aggregates will be different from the one mixed with rounded sand and crushed stones. Therefore, the way to achieve the ideal grading curve for each mixture of different aggregates is to involve the geometry of the aggregate particles by making use of particle packing models.

Equation 1 and Figure 1 describe the Fuller curve with q = 0.5 (Talbot and Richart, 1923), the curve should represent the aggregates gradation with the greatest density; however this empirically optimized gradation curve assumes particles of infinite finesses (i.e. $D_{min} = 0$). Based

on this assumption, the ideal particle distribution following with Fuller curve with q = 0.5 can never be fulfilled in practice.

$$P(D) = \left(\frac{D}{D_{max}}\right)^q$$
Eq. 1

Where, P(D): size cumulative distribution function (i.e. the fraction that can pass the sieve with opening D)

D: particle diameter being considered

D_{max}: the maximum particle size of the mix

q: parameter representing the distribution modulus, which adjusts the curve for fineness or coarseness



Figure 1. Ideal Packing curves according to Fuller, Andreasen and Funk and Dinger for D_{max} = 32 mm and D_{min} = 63 mm (Fennis 2012)

In 1968, Powers proposed another particle size distribution curve in which the power 0.5 is described as an exponent q in Equation 1. Based on the research of Andreasen and Andersen (1930), it is said that the voids content depends on the value of q and becomes direct ratio with

this distribution modulus, q. Considering of the inability of fine particles to pack in a similar manner as bigger but geometrically similar particles, Andreasen and Andersen proposed the use of an exponent q in the range of 0.33 - 0.50 (Hunger 2010). The values of q for achieving optimal packing density could be changed along with the packing density of individual size fractions and degree of compaction (De Larrard 1999a); hence, this adjustment factor should be determined experimentally with a consideration of the characteristic of the particles. For instance, q value of 0.45 is used in asphalt concrete mix design as a theoretical maximum packing density (Kennedy et al. 1994), while a q value of 0.4 is used by Hummel (1959) to achieve maximum packing density.

Aggregates are not infinite in reality; to avoid lean mixture, any real particle size distribution should have a finite lower size limit D_{min} (Funk and Dinger 1980). Therefore, based on Fuller curve and Andreasen's equation, Funk and Dinger (1994) modified it as Equation 2.

$$P(D) = \frac{D^{q} - D_{min}^{q}}{D_{max}^{q} - D_{min}^{q}}$$
Eq. 2

Where P(D) is the fraction of the total solids (percentage by volume) passing the particle size D; D_{min} and D_{max} denote minimum and maximum particle sizes, respectively, and exponent q is the distribution modulus. This distribution law delivers a feasible solution for a particle purpose. Mixtures with higher values of q is going to be coarser, whereas smaller q values results in finesrich granular blends as shown in Figure 2. Distribution modulus should be in a moderate range, too high or too low values of q affects the mixtures in a negative way. It is possible for a mixture with higher q value to have a high segregation potential and blocking, while a mixture with a lower value of q may have a high apparent viscosity because of the high amount of fines and dense packing. To verify or evaluate the packing effect theoretically, sum of the squares of the residuals is considered.



Figure 2. Modified A&A model with various q values (Wang 2014)

To verify or evaluate the packing effect theoretically, sum of the squares of the residuals (RSS) is considered. As the calculation equation shown as in Equation 3, the optimization of an actual mix is to minimize the RSS value to achieve target curve fitting.

$$RSS = \sum (P_{t-acutal} - P_t)^2$$
 Eq. 3

Where, Pt-actual: percentage passing each sieve (actual particle size distribution curve)

Pt: target percentage passing each corresponding sieve (A&A model curve)

The "ideal" curve should represented the grading with the greatest density and the optimum ingredients mixing proportion. Based on researchers' conclusions that the gradation that gives the greatest density of the aggregates alone may not give the greatest density necessarily when combined with water and cement due to the way of the cement particles fit into smaller pores (Fennis 2012). On the other hand, adjusting mixture proportion to a fixed optimization curve is relatively easier because it requires only a limited amount of input parameters; especially when the value of q is fixed, only the mixing particle size distributions of the available materials are necessary to be optimized. However, the shortage is that particle characteristics such as particle

shape are not taken into account, hence the output optimized particle size distribution based on model is not inevitably leads to mixture with the highest packing density. In the research of Palm and Wloter (2009) and Stroeven et al. (2003), it was shown that the application of gap graded mixtures can lead to higher packing densities.

Besides generating optimization curves based on packing modes, there are another two methods for particle optimization: analytical particle packing models and discrete element models. The analytical particle packing models calculate the overall packing density of a mixture in terms of the geometry of the combined particle groups; the discrete element models develop a "virtual" particle structure from a given particle size distribution to calculate the packing density of the mixture. However, there are limitations on discrete element models. The limitations in computational speed result in inability for concrete mixture optimization, because numerous mixtures have to be evaluated to find the optimal composition (Fennis 2012).

2.3 Particle packing and water demand

Particle packing density optimization of concrete mixtures provides positive influence for both fresh and hardened concrete properties. In a particle structure, addition of fine particles helps filling up the voids in the particle structure and leave minimum space for water. Therefore, addition of fine particles is one of the effective ways to reduce water demand (Kronlof 1997; Larrard 1999; Wong and Kwan 2008). A higher packing density leads to a smaller void ratio, therefore less amount of cement paste is need (Kwan and Mora 2001). Hence particle packing can improve concrete shrinkage and creep by providing a strong aggregate structure with a high packing density and reducing the water demand. It is known that the definition of particle packing density is the solid volume of particles in a unit volume. There should be a distinction made between the packing density of a stable particle structure and the volume of particles in a real concrete mixture. As shown in Figure 3, all particles are in contact with each other and packed with certain packing density in a stable particle structure (Fig. 3 b), while in a real concrete mixture (Fig. 3 a), the partial volume of all the particles in a unit volume. The same amount of particles in a stable particle structure is packed closer than in a real mixture, in other words, the density in a real concrete mixture is lower than the density in stable particle structure. For the real concrete mixture, part of the water is utilized to fill the voids between particles and react with cement, and the rest of the water is regarded as excess water. Excess water in concrete mixture provides flowability of the mixture. When the excess of water in the mix is higher, the flowability increases, and the solid content of the mixture decreases correspondingly.



Figure 3. The volume of a flowable mixture compared to the volume occupied by a stable particle structure containing the same particles (Fennis 2012)

In terms of the research of Fennis (2011), an increased packing density reduces the required amount of void water. In this way, concrete mixtures with the same workability can be designed with a lower water requirement, in other words, a lower water-to-cement ratio. Based on previous research, lower water-to-cement ratio contributes to a higher compressive strength (shown in Figure 4). Therefore, an increased packing density leads to the design of high strength concrete with a lower water-to-cement ratio or ecological concrete with a constant water-to-cement ratio but less amount of cement.



Figure 4. Experimental strength values vs. water/binder ratio (Yeh 1998)

2.4 Particle packing and rheology

Rheology can be termed as the study of the flow and deformation of materials whose flow properties are complicated in nature, rather than the fluids such as liquid or gas. Fresh concrete can be thought of as a fluid, and based on the research of Barnes in 1989, the basic rheology principles can be also applied to this material; hence, the concept of rheology can be utilized to analyze the properties of fresh concrete such as behavior of mix, deformation, and placement of concrete.

For the simplest fluid, it obeys Newton's law of viscous flow, and can be described as this equation: $\tau = \eta \dot{\gamma}$, where τ represents the shear stress (Pa), η is the coefficient of viscosity (Pa·s), and $\dot{\gamma}$ represents the shear rate (s⁻¹). Since fresh concrete is considered as a very concentrated suspension, a systematic investigation in the rheology of concrete has been carried out by Tattersall and Banfill in 1983. They stated that the fresh concrete flow could be described by the Bingham model: $\tau = \tau_0 + \eta \dot{\gamma}$, where τ_0 is the yield stress (Pa), which indicates the minimum stress to start a flow.

To ensure the workability of the concrete mortar, it requires a sufficient amount of cement paste to fill up the space between aggregates. The less void (space) between aggregates, the less paste required to fill up, therefore, there will be more paste coating the aggregates, which can improve the flowability of mortar. In figure 5, the flow curves of mortar with graded aggregates is below the curves of mortar with single-sized aggregates, which indicates the fact that graded aggregates improves the workability of mortar (Hu 2005).



Figure 5. Effect of sand gradation on mortar rheology (Hu 2005)

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Figure 6 shows the relationship between the rheological parameters and the fineness modulus of single-sized and graded aggregated, respectively. Compared with single-sized aggregates, graded aggregates leads to a lower yield stress and viscosity with a similar fineness modulus, because graded aggregates had less uncompacted void content, which results in less cement pasted needed to provide the same flow (Hu 2005).



Figure 6. Rheological parameters of mortar for graded and single-sized sand (Hu 2005)

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CHAPTER 3. INFLUENCE OF PARTICLE PACKING ON CONCRETE MORTAR WITH FIXED DISTRIBUTION MODULUS

Abstract

Optimum packing of mortar materials is one of the key components in mix design. Having optimum packing can lead to the beneficial effects of minimizing the amount of binder, denser aggregate distribution, reduction in creep and shrinkage and higher strength. Following an environmentally friendly approach, the present mortar mixtures utilizes industry by-products such as Class F fly ash, silica fume and limestone fines in combination with Type I/II Portland cement and river sand. The combination of these mortar materials are optimized by the modified Andreasen and Andersen (A&A) model for particle packing. Three types of mixtures are studied: (1) optimum proportion without changes in constituent material gradations, (2) modified limestone fine gradation, and (3) modified river sand gradation. The packing is verified by the value of the sum of the squares of the residuals, which can indicate how mixing curve fits the target curve, and the effects in fresh and hardened properties are investigated by the dry particles density, void ratio, rehology, and compress strength. The results indicate that enhanced particle packing improves mortar density and increases viscosity, but it had minimal effect on heat of hydration and yield stress of the mortar mixture.

3.1 Introduction

Concrete is a properly proportioned mixture consisted with cement, coarse and fine aggregates, water, supplementary cementitious materials (SCMs) and chemical admixture. In concrete mixture, exothermic reaction takes place between cementitious materials and water and the cementitious materials themselves, which generates heat and hydration products (such as CSH gel and Ca(OH)₂ crystal); this process, termed as hydration, leads to concrete harden and strength development. In the first few days of hydration, the generated heat of hydration may cause a considerable rise in concrete temperature, and the temperature will keeping rising over longer periods. With the heat temperature going up, the risk of shrinkage and crack will also increase, while in cold weather, heat of hydration could be used to help the concrete improve rate of hydration and avoid from freezing. Based on ACI (2013), the ultimate strength may be influenced by the initial rate of strength gain, and the initial or early rate of strength gain is directly correlated with the rate of hydration. With a faster early strength gain, the ultimate strength will become lower. Therefore, measurement of rate of hydration could provide an approximate idea about concrete strength.

Nowadays, researchers have been paid more and more attention to improving the properties of concrete. Addition of supplementary cementing materials (SCMs), such as fly ash, silica fume, and slag etc., is one of the efficient ways to improve concrete properties and performances. Besides that, optimizing aggregate grading is also an effective way for concrete improvement. Optimum particle packing provides lower porosity by minimizing the amount of fine particles needed to fill up the space between aggregate particles; on the other hand, for binder system, optimum packing of binder particles will improve binder flowability by deducting the water demand for filling the space between powder particles. In 2010, Hunger suggested that the mixture of solids in fine and

coarse sections should be optimized separately due to the fine particle fractions primarily contribute to the mixture porosity. Denser fine aggregate packing provides a denser microstructure and an increase of contacts between particles for continuity of load transfer resulting in the better compressive strength, workability, and watertightness which improve the chemical and frost resistance under sufficient content (Korjakins 2013). In this chapter, a selected concrete mortar mixture will be optimized by modified A&A model, and by performing a series of tests on fresh and hardened concrete mortar, the influence of particle packing on flow and strength of concrete mortar can be studied.

3.2 Materials and Mix Proportion

For commonly concrete used in field, the mixture content usually includes Portland cement, water, fine and coarse aggregate, chemical admixture, and SCMs, such as fly ash and silica fume. In this study, Type I Portland cement, river sand, limestone fines, class F fly ash, and silica fume are selected as concrete mortar materials. Figure 7 shows the particle size distributions of those solid fine materials.



Figure 7. Particle size distribution of portland cement, limestone fines, river sand, silica fume, and fly ash

Considering the development for HPM, four binder mixtures with different amount of fly ash and silica fume for calorimetery test are listed in Table 1 (proportions by mass). With the constant mix proportion of cement, limestone fines, water, and superpasticizer, the amount of fly ash is decreased from mixture 1 to 4, while the amount of silica fume is increasing. Both silica fume and class F fly ash are pozzolanic materials, which can take place part of Portland cement and benefit on fresh and hardened concrete. For instance, addition of fly ash can reduce heat of hydration evolution, improve workability and increase long-term compressive strength; silica fume added to concrete improves concrete durability and also increases its early and ultimate compressive strength.

%	Mixture 1	Mixture 2	Mixture 3	Mixture 4
Cement	36.0	36.0	36.0	36.0
Fly ash	10.8	8.4	6.1	3.8
Silica fume	0.0	2.4	4.7	7.0
Limestone fines	30.4	30.4	30.4	30.4
Water	18.4	18.4	18.4	18.4
Superplasticizer	4.4	4.4	4.4	4.4
Total	1	1	1	1

Table 1. Mix proportion (%) of binder for calorimetery test

For the binder mixtures, the water to binder ratio is 0.24. Considering the sufficient lubrication between particles, dispersants such as high range water reducer (HRWR) can be used to help these binder particles avoid agglomerating.

3.3 Test Procedures and Equipment

3.3.1 Heat of hydration

Isothermal calorimetry is utilized to measure the rate of heat of hydration of the four binder mixtures, which provides information on the various exothermic reactions between water and cementitious materials and the cementitious materials themselves. During the process of heat generation, rate of reaction and reactivity can also be reflected.

The equipment of isothermal calorimetry is shown in Figure 8. The isothermal calorimeter contains 8 channels in a temperature control chamber, and each channel measures heat flow from an individual specimen independently. Once specimens are placed in the calorimeter, the heat generated by the specimens will flow to the aluminum sample holder and then towards a heat flow detector (Wang 2016).



Figure 8. Isothermal calorimeter

To measure the rate of heat of hydration for a given paste or mortar, 50 grams of dry materials and corresponding amount of water with admixtures were prepared separately for each measurement. Solid dry and liquid materials were placed in separate plastic cups and then placed in the isothermal calorimeter for 24 hours for conditioning. This step aims to make sure all samples in the chamber reach the designed initial temperature (20°C) before mixing. After conditioning, the dry and liquid samples were taken out from the chamber, mixed, and put back quickly into the chamber (Wang 2016). When the at least 48-hour data record was completed, the rate of heat generation in mW per gram of cement (mW/g) was calculated by using Equation 4:

$$P = \frac{(R-B)CF}{ws/(1+w/c)}$$
Eq. 4

Where, R: calorimeter data reading, mV

B: calibrated base line, mV
CF: calibration factor, mW/mV (ranged from 14.21 to 16.16 mW/mV)
ws: mass of sample, g
c: mass of cementitious materials, g
w: mass of water, g

3.3.2 Particle packing analysis

The particle packing model used for analysis is the modified Andreasen & Anderson (A&A) model (Funk and Dinger 1994) shown as Equation 5, where P(D) is the fraction of the total solids (percentage by volume) passing the particle size D; D_{min} and D_{max} denote minimum and maximum particle sizes, and exponent q is the distribution modulus.

$$P(D) = \frac{D^q - D_{min}^{\ q}}{D_{max}^{\ q} - D_{min}^{\ q}}$$
Eq. 5

The distribution modulus, q, has a recommended range from 0.33 to 0.50 (Hunger 2010). With an increasing value of q, the ideal packing distribution is going to become coarser; likewise, a decreasing value of q results in a finer-sized particles in the ideal packing distribution. In this chapter, a fixed distribution modulus value of 0.25 was used for entire packing analysis. With the fixed q value, the ideal particle packing distribution was determined for the optimizing of unmodified mortar mixture.

For packing analysis, the sum of the squares of the residuals (RSS) was introduced and used to describe how the actual grading fits the target curve. The equation of RSS is expressed in Equation 6. The actual grading curve can be optimized by minimizing RSS value. Since this analysis focused on concrete mortar, the binder mixture were mixed with river sand with a weight ratio of 30:70.

$$RSS = \sum (P_{t-acutal} - P_t)^2$$
 Eq. 6

Where, P_{t-actual}: percentage passing each sieve (actual particle size distribution curve)

Pt: target percentage passing each corresponding sieve (A&A model curve)

3.3.3 Packing density

Dry density of mortar materials was prepared using the equipment as shown in Figure 9. The tested fine materials were placed into a 100 x 100 x 150 mm rigid box in three layers of equal height. Each layer was consolidated by placing a weight that exerts a consolidation pressure of 4.1 kPa and then vibrating for 1 minute.



Figure 9. Equipment for consolidation for specimen preparation (Lomboy 2012)

After the consolidation, the height at four corners was measured by a caliper, and the weight of specimen was also weighted; then the dry density and void ratio were calculated by Equation 7 and 8.

$$\rho = Mass / Voulume of material$$
 Eq.7

3.3.4 Flowability and rheology

To measure the flowability of concrete mortar, mini slump cone test was performed by using a smooth flat plate and a flow mold described in ASTM C230 (Figure 10). Before testing, the plate and mold should be moistened. Place the moistened mold on the center of the plate, and then the freshly mixed concrete mortar was poured into the mold with tamping. After leveling the top surface of the mold and cleaning the sides of the mold, lift the mold slowly and vertically. The diameter measured by taking the average of two perpendicular directions were obtained until the spread of the flowing mortar on the plate stopped.



Figure 10. Mold and smooth plate for flowability test

The rheological properties test was conducted utilizing the Brookfield rheometer device (Figure 11) to measure viscosity, thixotropy, and yield stress. To performing the test, the desired shear stress speed was selected, and the freshly mixed concrete mortar was poured into a 50 mm diameter x 100 mm height sample cylinder. Equip the sample cylinder onto the rheometer with a small spindle immersed inside the mortar along the axle wire of the cylinder. The rheology test firstly runs 60 seconds up-curve with shear rate increasing from 0 s⁻¹ to 100 s⁻¹, and then runs 60

seconds down-curve with shear rate decreasing to 0 s^{-1} . The relation between shear stress and shear strain were plotted and displayed by the Brookfield automatically. Viscosity, yield stress, and thixotrophy of sample was calculated based on the plot.



Figure 11. Equipment for rheology test (Wang 2012)

3.3.5 Compressive strength

Concrete mortar was casted in 2-inch cubic sample with a water-to-binder ratio (w/b) of 0.46. The compressive strength of the cubic samples was measured in 1, 3, 7, and 28 days of age. In each of the strength measurement ages, three samples of each mix were tested and the average of the three measurements were presented as the corresponding compressive strength. The average compressive strength values were plotted versus ages for investigation.

3.4 Results and Discussion

3.4.1 Material optimization

Four groups of binder mixtures listed in Table 1 were conducted isothermal calorimetery test, and the curve of rate of heat generation versus time was plotted in Figure 12.



Figure 12. Plot of rate of heat generation vs. time for four binder mixtures

Based on the plotted result, there is no big difference for their rate of reaction and reactivity. Sample 1, 2, and 4 have the approximately equal rate of heat generation, while sample 3 obtained the highest rate of heat generation, which indicates higher strength gain. Therefore, sample 3 was selected as the original HPM mix for subsequent packing analysis.

3.4.2 Packing analysis

To perform the concrete mortar particle packing analysis, the selected original HPM mix was mixed with river sand following a weight ratio of 30:70 to consist the unmodified mix. Then two types of modification were made for optimizing: modified limestone fines gradation (LS modified) and modified river sand gradation (RS modified). Based on A&A model, gradations of these three mixes and the target "ideal" gradation curve were plotted in Figure 13, and the value

of sum of the squares of the residuals (RSS) was listed in Table 2 to verify the packing results with a fixed distribution modulus of 0.25.



Figure 13. Particle size distribution for mortar mixes

For LS modified mix, limestone fines gradation was modified by adding limestone fines retaining on No. 100 sieve (149 um), No. 140 sieve (105 um), and No. 200 sieve (74 um), respectively, without changing the total proportion occupied by limestone fines. For RS modified mix, river sand with particle size smaller than No. 50 sieve (297 um) was added into the original mix without changing the weight ratio of river sand. With the fixed q value, RS modified mix is the closest to the target curve, and the LS modified mix is the farthest one.

Table 2. RSS value of unmodified, LS modified, and RS modified mix.

	Unmodified	LS modified	RS modified
RSS	1207	2618	387

According to RSS value, LS modified mix obtained the highest RSS value indicating a poor match with the target curve; while RS modified mix had a good packing result with the lowest RSS value of 387. The way of modifying particle gradation of limestone fines was failed achieve the goal of optimizing with q = 0.25. So in terms of RSS value and the packing model, RS modified mix with the lowest RSS value was regarded as the optimum packing, and LS modified mix can be treated as the worst case with poor particle packing gradation. All of these three mixes were compared to investigate the effect of particle packing on fresh and hardened properties of concrete mortar.

3.4.3 Dry density and void ratio

Based on the packing gradation of three mixes, ingredients of unmodified, LS modified, and RS modified mixes were prepared and mixed separately to measure the bulk density and calculate corresponding voids ratio. Figure 14 shows the dry density and calculated void ratio results of unmodified, LS modified, and RS modified mixes.



Figure 14. (a) Dry density results; (b) Calculated void ratio results

Unmodified mix obtained the highest dry density corresponding to the lowest void ratio; likewise, RS modified mix with the lowest dry density has the highest void ratio. Related with RSS value, it was shown that higher RSS value results in lower dry density; meanwhile, lower RSS value leads to lower void ratio.

3.4.4 Fresh properties

3.4.4.1 Flowability

Mini slump cone test was conducted to investigate the flowability of unmodified, LS modified, and RS modified mixes. The same flow diameter of 4.5 inches and the same slump height of 1.27 inches were obtained. Therefore, particle packing do not have a significant influence on flowability measured by mini slump con test.

3.4.4.2 Rheology

Rheology test provides information of viscosity and yield stress of concrete mortar. Figure 15 (a) shows the loading history of the test, and Figure 15 (b) shows the flow curves of unmodified, LS modified, and RS modified mixes. The flow curve displayed as a loop, and the down curve follows Bingham model $\sigma = \sigma_0 + \eta \dot{\gamma}$ to determine viscosity η and yield stress σ_0 . Rheology test was conducted using two samples for each type of mix; detailed flow curve of each sample was shown in Appendix.





Figure 15. (a) Loading history of rheology test; (b) flow curves of three mixes

The viscosity was calculated as the slope of the down flow curve from the shear rate range from 20 s⁻¹ to 80 s⁻¹, and the corresponding yield stress was determined as the intercept of the straight line with the same slope. Figure 16 shows the calculated viscosity and corresponding yield



stress. According to the viscosity results, viscosity of concrete mortar increased with density. No trend observed from the bar chart of unmodified, LS modified, and RS modified mixes.

Figure 16. (a) Viscosity results; (b) yield stress results

3.4.5 Compressive strength

The strength ages of 1, 3, 7, and 28 days were investigated for unmodified, LS modified, and RS modified mixes. According to Figure 17, there is a good trend of 1-day compressive strength along with the corresponding dry density. For early strength, unmodified mix with the highest density had a relative high compressive strength; however, the density (or particle packing) has no significant influence on 28-day strength. LS modified mix had an ultra-high 28-day strength, which may because of the reaction between limestone fines and fly ash.



Figure 17. Compressive strength data

3.5 Observations

Through modified A&A model, three types of mixes: original mix (unmodified mix), modified limestone fines gradation mix (LS modified), and modified river sand gradation mix (RS modified) were prepared to test their dry density, void ratio, flowability, rheology, and compressive strength. Based on the results obtained, following observations can be drawn from the present study:

- The trend of RSS calculated is not consistent with that of dry density. With the fixed distribution modulus, lowest RSS value obtained the smallest dry density, while the highest RSS value had a moderate one.
- Particle packing did not influence the mortar flow values measured by mini slump cone test, because that samples with different RSS value all obtained the same flowing diameter.
- Particle packing improves workability of concrete mortar. Viscosity of concrete mortar did not have consistency with RSS, but it increased with density increasing. Denser mixture results in more particle interlock and friction, which increases viscosity. The mixture with optimum packing obtained higher workability.
- The specimen with high packing density has a higher compression strength. The trend of 28-day compressive strength is not consistent with the trend of density considering with the disturbance of the addition of SCMs.

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CHAPTER 4. EFFECT OF PARTICLE PACKING WITH DIFFERENT DISTRIBUITON MODULUSON CONCRETE MORTAR

Abstract

Concrete mix gradation with optimum packing leads to the beneficial effects of minimizing the amount of binder, denser aggregate distribution, reduction in creep and shrinkage and higher strength. Various distribution modulus values (q = 0.25, 0.35, and 0.45) were used to evaluate the particle packing quality of a given high performance mortar (HPM). Single sized sand was selected and added to the HPM mixture so as to make the PSD of the modified HPM mixture to the A&A model curve. The minimum sum of squares of the residuals (RSS) was used to assess the quality of the PSD modification. Dry density, rheology, and compressive strength tests were performed for the both original mortar mix and sand-modified mixes. The results show that the increasing q value decreased mortar density, viscosity, and early age strength but had little/no effect on 28-day mortar strength.

4.1 Introduction

Optimization curves is one of the efficient particle optimization methods, which set a specific particle size distribution (or called "ideal" gradation curve) to let groups of particles combine in such a way that the total particle size distribution of the mixture is closest to an optimum curve (Fennis 2012). The modified Andreasen & Anderson (A&A) model (Funk and Dinger 1994) is a popular mathematic model generating the optimization curves for particle packin. This continuous packing distribution model is expressed as the volume percentage of particles (P_t) passing particle size D with D_{min} and D_{max} denoting minimum and maximum particle sizes, which is shown as equation: $P(D) = \frac{D^q - D_{min}^q}{D_{max}^q - D_{min}^q}$. In 1930, Andreasen and Andersen stated that the void content only depends on the value of distribution modulus, q; they also limited the q value of a range from 0.33 to 0.50 (Hunger 2010). Additionally, distribution modulus controls the fineness of the generated mix as well. With an increasing value of q, an ideal packing distribution for particles will have a greater number of larger-sized particles; on the contrary, smaller q generates an ideal packing distribution with more smaller-sized particles (Wang 2016). It was found that the values of the distribution modulus for optimizing particle packing varied with individual size fractions and the degree of compaction (De Larrard 1999a). Therefore, in the present study of this chapter, the effect of particle packing with three different distribution modulus (q = 0.25, 0.35, and0.45) on HPM was discussed by conducting series of tests, such as dry density test, rheology test, and compressive strength test.

4.2 Materials and Mix Proportion

In the study of this chapter, no supplymentery cementitious materials (SCMs) were used to take place part of Portland cement. The addition of SCMs benefits concrete in many ways. For instance, addition of fly ash reduces the heat generation during hydration process and increases long-term strength of concrete. However, to show how the different values of distribution modulus influence concrete mortar properties, only type I/II Portland cement and river sand with a weight ratio of 30:70 was utilized as sample mix. Figure 18 plots the particle size distribution (PSD) of type I/II Portland cement and river sand.



Figure 18. Particle size distribution of Portland cement and river sand

This figure indicates that, with the same gradation sieve size, the particle size distribution for cement is much finer than the particle size distribution of river sand. Considering the performance of HPM, a water-to-cement (w/c) of 0.28 was designed to gain higher strength. To

ensure the sufficient lubrication between particles, dispersants such as high range water reducer (HRWR) with a dosage of 15 oz/cwt can be used to help these binder particles avoid agglomerating.

4.3 Test Procedures and Equipment

4.3.1 Particle packing analysis

The particle packing model used for analysis is the modified Andreasen & Anderson (A&A) model (Funk and Dinger 1994) expressed as Equation 9, where P(D) is the volume percentage of solids passing the particle size D; D_{min} and D_{max} denote minimum and maximum particle sizes, and exponent q is the distribution modulus.

$$P(D) = \frac{D^q - D_{min}{}^q}{D_{max}{}^q - D_{min}{}^q}$$
Eq. 9

The distribution modulus, q, has a recommended range from 0.33 to 0.50 (Hunger 2010). With an increasing value of q, the ideal packing distribution is going to become coarser; likewise, a decreasing value of q results in a finer-sized particles in the ideal packing distribution. In this chapter, a distribution modulus, q, was set to different values, namely, 0.25, 0.35, and 0.45. The ratio (by mass) of cement to river sand was selected as 30: 70, and the optimum mix gradations for each q was determined accordingly. The optimum mix gradation was verified based on the minimum sum of squares of the residuals (RSS). The equation of RSS is shown as follow:

$$RSS = \sum (P_{t-acutal} - P_t)^2$$
Eq. 10

Where, P_{t-actual}: percentage passing each sieve (actual particle size distribution curve)

Pt: target percentage passing each corresponding sieve (A&A model curve)

4.3.2 Packing density

The procedure and equipment of dry density test in this chapter was the same as that in Chapter III. The dry mixed sample were placed into a 100 x 100 x 150 mm rigid box in three layers of equal height. Each layer was consolidated by placing a weight that exerts a consolidation pressure of 4.1 kPa and then vibrating for 1 minute. After the consolidation, the height at four corners was measured by a caliper, and the weight of specimen was also weighted; then the bulk density and void ratio were calculated by following equations:

$$\rho = Mass / Voulume of material$$
 Eq.11

4.3.3 Rheology

The rheological properties test was conducted in the same way with the rheology test in Chapter III. The freshly mixed concrete mortar was poured in to a sample cylinder with a dimension of 50 mm diameter x 100 mm height. Then the sample cylinder was equipped on the Brookfield rheometer device with a small spindle immersed inside the mortar along the axle wire of the cylinder (Figure 19). Under a selected shear stress speed, the device started to measure viscosity, thixotropy, and yield stress. The rheology test firstly runs 60 seconds up-curve with shear rate increasing from 0 s⁻¹ to 100 s⁻¹, and then runs 60 seconds down-curve with shear rate decreasing to 0 s⁻¹. In each rheology measurement, 2 samples of each mix were tested, and the relation between shear stress and shear strain were plotted and displayed by the Brookfield automatically. Viscosity, yield stress, and thixotrophy of sample was calculated based on the plot.



Figure 19. Equipment for rheology test (Wang 2012)

4.3.4 Compressive strength

Concrete mortar was casted in 2-inch cubic sample with a water-to-binder ratio (w/b) of 0.28. The compressive strength of the cubic samples was measured in 1, 3, 7, and 28 days of age. In each of the strength measurement ages, three samples of each mix were tested and the average of the three measurements were presented as the corresponding compressive strength. The average compressive strength values were plotted versus ages for investigation.

4.4 Results and Discussion

4.4.1 Packing analysis

The gradation of cement and river sand were provided, and it was expected to develop a mixed gradation curve getting as close as possible to the target gradation curve for each of the different values of distribution modulus. The minimum and maximum particle sizes were set to 0.399 μ m and 4570 μ m respectively. The particle size distributions of target curve and the actual mix curve (before and after optimizing) for each of different q values were plotted in Figure 20, 21, 22, and the RSS values were list in Table 3.



Figure 20. Particle size distribution for q = 0.25



Figure 21. Particle size distribution for q = 0.35



Figure 22. Particle size distribution for q = 0.45

Table 3. RSS value for original and optimum mix with q = 0.25, 0.35, and 0.45

	q = 0.25		q = 0.35		q = 0.45	
	Original Mix	Optimum Mix	Original Mix	Optimum Mix	Original Mix	Optimum Mix
RSS	1348	310	1662	1326	4960	3786

Original mix curve obtained a better match with the target curve with q= 0.25. The target curve is moving rightward as the value of distribution modulus increasing, in other words, the target gradation is coarser with a larger value of distribution modulus. For original mix optimizing, several sieve sizes of No. 4, No. 8, No. 16, No. 30, No. 50, and No. 100 were utilized to re-grading the river sand. The graded river sand with new particle size distribution was generated and then combined with cement to achieve an optimum mix curve.

4.4.2 Dry density and void ratio

Three optimized dry mixes with different values of distribution modulus of 0.25, 0.35, and 0.45 based on modified A&A model were tested for their dry density. Table 4 listed the test results of dry density and calculated void ratio for three optimized dry mixes, and the relationship for the distribution modulus, dry density and calculated void ratio was described in Figure 23 and 24. *Table 4. Test results of dry density and void ratio of three optimized mixes*

	q = 0.25	q = 0.35	q = 0.45
Dry density (g/cm^3)	1.875	1.844	1.826
Void ratio (%)	0.579	0.631	0.653



Figure 23. Plot of distribution modulus vs. dry density



Figure 24. Plot of distribution modulus vs. void ratio

The target curve provides a way for actual mix to be optimized by getting as close as possible to it (i.e. reducing RSS value). However, this target curve is not unique for all mixtures. The target curve is various with the changing of q value. For instance, q value of 0.45 is used in asphalt concrete mix design as a theoretical maximum packing density (Kennedy et al. 1994), while a q value of 0.4 is used by Hummel (1959) to achieve maximum packing density. For the mixture in this chapter, target curve with q of 0.25 is the best one for matching. Although the original mix was also optimized based on A&A model with q of 0.35 and 0.45, the most matching one brought a highest dry density. So it could say for the same mixture, increasing q value leads to coarser mixture, and the same consistency works for smaller q. For the mixture with more fine particles, larger q value results in a lower dry density and higher void ratio.

4.4.3 Rheology

In this test, totally six samples for three actual optimization mix with q of 0.25, 0.35, and 0.45 were tested for rheological properties. For each actual optimization mix, two samples were tested and the average result of viscosity and yield stress was performed. The flow curves for all three mixes were drawn in Figure 25. The viscosity of different q values was plotted in Figure 26.



Figure 25. Flow curve for three mixes with q = 0.25, 0.35, and 0.45



Figure 26. Viscosity for three mixes with q = 0.25, 0.35, and 0.45

The yield stress is zero for all of three mixes due to the use of HRWR with a dosage of 15 oz/cwt. According to the test results, the viscosity is decreasing when q increases with a given

water-to-cement ratio. For a given proportion, lower q leads to finer-particle in concrete mortar mix, which increases the viscosity of mortar. The surface area results in less paste thickness to coat the aggregate and provide flow.

4.4.4 Compressive strength

Compressive strength test results of three mortars based on modified A&A model with different values of distribution modulus are shown in Figure 27. For 1-day strength, there is a clear increasing trend as q increases; however for the rest days of strength, no obvious trend was found. The results were not as good as expected, especially the 7-day strength was higher than 28-day strength for all three types of mortar, so we decided to repeat 7-day and 28-day strength test, and the results are shown in Figure 28.



Figure 27. Compressive strength data for three types of mortar with q = 0.25, 0.35, and 0.45



Figure 28. Repeated compressive strength data for three types of mortar with q=0.25, 0.35, and 0.45

For 28-day strength, the mortar with q=0.45 obtained the highest strength for both original and repeated batches, while 7-day strength had a decreasing trend as q increases. In terms of the testing results, it can be stated that a lower value of q leads to a relatively higher early age strength. Generally, with a given water-to-cement ratio and cement-to-aggregate ratio, there are still many factors affecting concrete compressive strength, such as quality of raw materials, compaction of concrete, and curing of concrete etc. On the other hand, the target packing curve gives the optimum density of the dry mixture alone may not necessarily give the optimum density when mixed with water because of the way the cement particles fit into smaller pores (Fennis 2012).

4.5 Observation

The mortar with the constant w/c and cement to aggregate ratio was optimized by the target curve with different values of distribution modulus was conducted dry density test, rheology test, and compressive strength test. Based on the test results, the following observations was found:

- Distribution modulus controls the characters of particles. Increasing q value results in a coarser particle size distribution; on the contrary, the gradation curve with smaller q value is getting finer.
- Rheological properties of concrete mortar and q value have an inverse relationship. Since larger q value leads to coarser mixture, which also results in a decrease in viscosity and an increase in workability.
- Early age compressive strength is influenced by the changing of distribution modulus: lower value of q results in a higher early age strength.

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CHAPTER 5. CONCLUSIONS AND RECOMMENDATIONS

The purpose of this study is to investigate the effect of particle packing on flow property and strength of concrete mortar. For the entire study, modified A&A model was selected as the scientific approach to provide an "ideal" particle packing distribution, which should obtain the optimum particle proportion and lead to optimum packing density. The study was separated into two parts. Part 1 focused on the influence of particle packing with fixed distribution modulus on flow property and strength of concrete mortar. In the study of part 1, the mortar material of type I/II Portland cement, limestone fines, Class F fly ash, silica fume, and river sand were considered. For part 2, the influence of particle packing with different values of distribution modulus on flow property and strength of concrete mortar was investigated. Type I/II Portland cement was used as the cement, and river sand was used as fine aggregate. Based on the test data and results, following conclusions and recommendations can be drawn:

<u>Part 1</u>

- The trend of RSS calculated is not consistent with that of dry density. With the fixed distribution modulus, lowest RSS value obtained the smallest dry density, while the highest RSS value had a moderate one. Since the way the cement fits into the small pores, the mixture optimized by target packing curve is not necessarily obtained the highest density when mixed with water. Therefore, the measurement of air voids and density after mixing would help study packing influence.
- Mortar flowability is not influenced by particle packing based on modified A&A model.

- Particle packing improves workability of concrete mortar. Viscosity of concrete mortar did not have consistency with RSS, but it increased with density increasing. Denser mixture results in more particle interlock and friction, which increases viscosity. The mixture with optimum packing obtained higher workability.
- The specimen with high packing density has a higher compression strength. The trend of 28-day compressive strength is not consistent with the trend of density, hence, a further study about the packing influence on long-term strength is suggested to be developed.

<u>Part 2</u>

- Distribution modulus controls the characters of particles. Increasing q value results in a coarser particle size distribution; on the contrary, the gradation curve with smaller q value is getting finer.
- Rheological properties of concrete mortar is influenced by q values. Larger q value leads to coarser mixture, which also results in a decrease in viscosity and an increase in workability.
- For fine-rich granular blends, higher q value leads to a lower early age compressive strength of concrete mortar. Additionally, more different samples with different material proportion are expected to prepared for further study; meanwhile, the long-term strength should also be considered in the future.



APPENDIX: FLOW CURVES FOR ALL TESTING SAMPLES









Shear Rate,1/s



