

Effects of PSD and surface morphology of micro-aggregates on admixture requirement and mechanical performance of micro-concrete

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Abstract

Micro-concrete (MC) can be defined as a high performance cement-based material proportioned with micro-aggregates whose particle sizes range from about 0.5 mm to less than 1 μm . The incorporation of micro-aggregates improve the particle packing density of the cementitious system, by this way the rheological and mechanical properties of MCs are enhanced.

An experimental program has been conducted to investigate the effect of three types of micro-aggregates presenting three different particle size distributions (PSDs) on the admixture requirement, compressive and flexural strength development of MC. In order to compare the PSD of micro-aggregates and to find a correlation between fineness and admixture requirement, the concept of “Fineness Index” was described. Additionally, particle shape and surface morphology of micro-aggregates were analysed by using SEM images. Some conventional commands of image analysis were employed. Possible quantification methods of shape characteristics and surface roughness of micro-aggregates were proposed. The results showed that, in addition to PSD, particle shape and surface morphology of micro-aggregates should also be quantitatively determined. Success of characterization significantly depends on preparation of appropriate and representative SEM images and proper selection of methodology of analysis.

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Keywords: Micro-concrete; Micro-aggregate; PSD; Surface morphology; Admixture; Fineness index; Image analysis

1. Introduction

The development of high-performance cement-based materials like self-compacting concrete (SCC) and reactive powder concrete (RPC) have increased the research on micro-aggregates coupled with the effects of superplasticizers. Mixture proportioning of powder-type SCC and RPC requires high amounts of fine ground materials. While these fine ground materials improve the particle packing density of cementitious system, the superplasticizers help to obtain the desired rheological properties by increasing the workability without causing segregation in fresh state and improve the mechanical properties and durability by

reducing the water/cement ratio [1,2]. Some of the above mentioned fine powder materials are either industrial by-products or unprocessed materials. They provide environmental relief because industrial by-products are being recycled, hazardous emissions released into the atmosphere due to cement production are reduced, raw materials are preserved, and energy is saved [3,4]. Besides, semi-inert micro-aggregates such as finely ground limestone or quartz can be alternatively employed for high performance concrete mix design purposes. More recent work [5] has addressed the effects of micro-aggregates on rheological properties of high strength concrete. In the future, if feasibility and technical suitability are verified, the tendency of increasing the amount of micro-aggregates in concrete system may eliminate the use of coarse aggregate and sand.

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Micro-concrete (MC) can be defined as a high performance cement-based material proportioned with micro-aggregates whose particle sizes range from about 0.5 mm to less than 1 μm . MC is a matrix phase including all fines (cement, pozzolanes and fines in aggregates), water and possible chemical admixtures. In order to obtain an effective MC mix-design, the particle size distribution (PSD) of various micro-aggregates should be optimised to obtain maximum compactness. A full three-dimensional model or simulation of such a perfect packing system is still far from being realized because of the difficulties to deal with the large amount of very small particles [6]. The related works generally classified the factors that affect the matrix compactness into four groups: particle morphology, particle packing, interparticle spacing and matrix rheology [7]. Detailed research reports related with these topics can be found in the literature [8–20]. However, few studies have been focused on the multiple effects of micro-aggregate shape and surface texture in relation with PSD change. In this paper, the effects of micro-aggregate type (particle shape and surface morphology) and PSD on the admixture requirement and strength of MC have been investigated. The particle shape and surface morphology of micro-aggregates were analysed by using SEM images. Some conventional commands of image analysis were employed. Possible quantification methods of characterization of micro-aggregates were proposed.

2. Experimental

Experimental program has been divided into three parts: (i) Characterization of materials, in particular micro-aggregates, (ii) preparation of MCs and determination of admixture requirement for constant consistency, and (iii) determination of mechanical properties.

2.1. Materials

An ASTM Type I cement (CEM I 42.5) and three types of micro-aggregates at nine different PSDs were used. A high purity and fine non-densified silica fume was also employed. PSD of cement by LASER Diffraction Scattering (LDS) Analysis is plotted in Fig. 1. It was not possible to determine the PSD of silica fume. An example of theoretical calculated PSD of silica fume based on assumptions (particle size distribution is supposed to be bilinear) given by Larrard and Sedran [12] is also plotted in Fig. 1. Micro-aggregates at different PSDs were prepared by grinding. Each micro-aggregate has been described by a two-component code designation: the letters reflecting micro-aggregate type as quartz (Q), limestone (L) and river sand (R), followed by the lowercase letters (c), (m) and (f) reflecting coarse, medium and fine grinding, respectively. Detailed characterization of micro-aggregates will be presented in the subsequent section. The chemical analyses of cement, silica fume and micro-aggregates are given in Table 1.

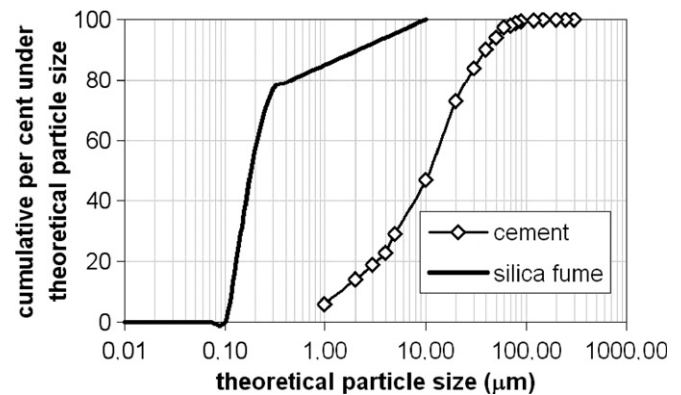


Fig. 1. PSD of cement by LDS analysis and theoretically calculated PSD of silica fume.

Table 1

Chemical analysis of Portland cement, silica fume and micro-aggregates

Chemical composition (%)	Portland cement	Silica fume	Micro-aggregates	
			Quartz (Q)	Limestone (L)
SiO ₂	20.40	89.30	99.5	0.55
Al ₂ O ₃	4.82	3.75	–	0.17
Fe ₂ O ₃	2.95	0.90	–	0.15
CaO	63.86	–	–	57.6
MgO	1.52	2.10	–	0.60
SO ₃	2.05	0.35	–	–
Cl	0.037	–	–	–

A polycarboxylate type high-range water reducing admixture complying with the ASTM C 494 (Type F) specification [21] was used in all mixes. The solid content, pH and specific gravity of admixture were 35.7%, 6.5% and 1.11%, respectively.

2.2. Characterization of micro-aggregates

2.2.1. PSD and fineness index concept

In this study PSDs of micro-aggregates were tested at Malvern model laser particle size analyser by using the Laser Diffraction Scattering Method (LDS) [22–24]. The PSDs of micro-aggregates of quartz, river sand and limestone are plotted in Fig. 2a–c, respectively. PSD is not a sole number that can be used for mix proportioning design of MC. The concept of “Fineness Index (FI)” containing both surface area and volume parameters in its structure can be used for mixture proportioning of MC. “Fineness Index (FI)” of micro-aggregates were calculated by using the results derived from PSD tests. The theoretical surface to-volume ratio of each definite average particle size (300, 250, 200, 150, 120, 90, 80, 70, 60, 50, 40, 30, 20, 10 and 5 μm) is multiplied by the passing percentages of each definite average particle size and the sum is called FI:

$$\text{FI} (\mu\text{m}^2/\mu\text{m}^3) = \sum_{i_{\text{definite}}=5}^{300} \frac{A_{i_{\text{definite}}}}{V_{i_{\text{definite}}}} p_{i_{\text{definite}}}$$

where i is the average particle size of the selected particle diameter interval, A_i is the single surface area of definite

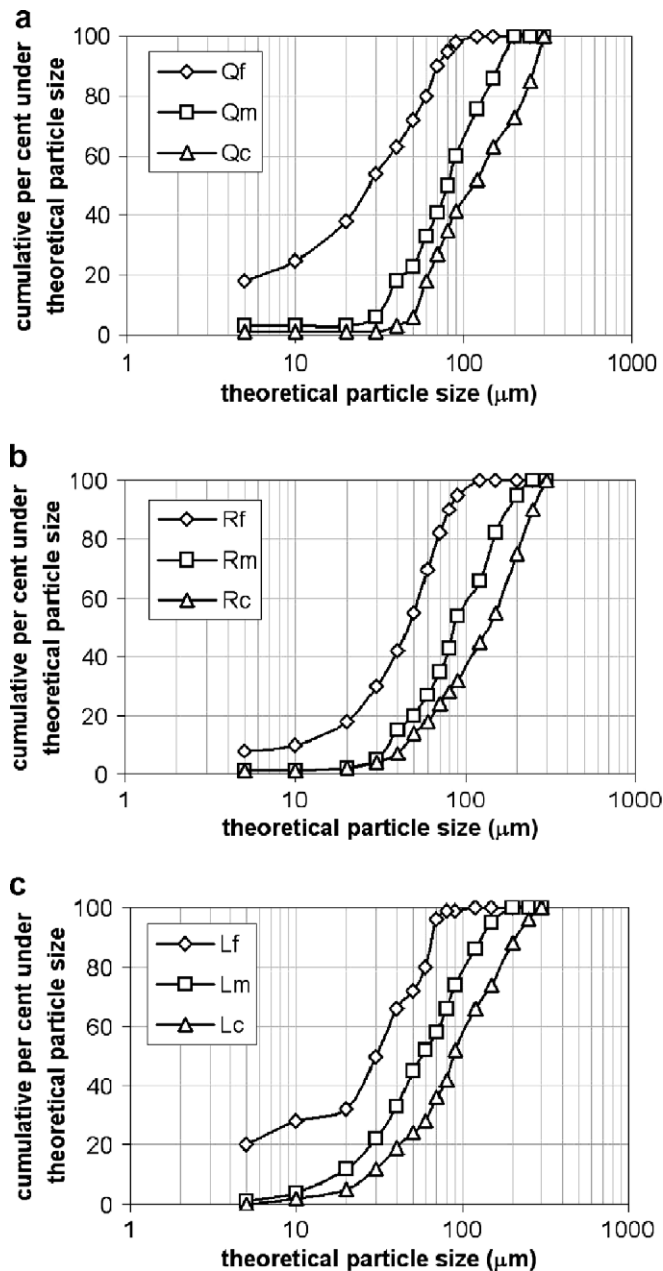


Fig. 2. Theoretical particle size distribution of micro-aggregates. (From LDS analyses, assuming all particles are spherical.)

average particle size of i , V_i is the single volume of definite average particle size of i , p_i is the cumulative passing percentage of definite average particle size of i . FI can also be described as cumulative surface area of unit volume of

particles of all selected theoretical average particle sizes. The calculated FI of micro-aggregates are listed in Table 2.

2.2.2. Particle shape and surface texture characterization of micro-aggregates from SEM images

The fine and coarse ground micro-aggregates of each type were examined by using SEM images. Secondary electron images were captured. The same amounts of micro-aggregates were poured and the same gently pressing procedure has been applied on to the carbon band. After sample preparation, SEM images of each type of micro-aggregates were captured at both low (30×) and high (250×) magnifications. The maximum areas of captured images were approximately 12.8 mm² and 0.16 mm² at low and high magnifications, respectively. Representative images of high (250×) magnifications were selected and are presented in Figs. 3–5. It should be noted that in case of low magnification, the concentration of micro-aggregates varies from place to place. By grinding this situation is more valid for micro-aggregates of river sand and limestone, while a more homogeneous dispersion was obtained from the micro-aggregates of quartz. Note that the ungrounded raw condition of micro-aggregates of quartz is presented in Fig. 3a for comparison purpose. This very coarse quartz was not used in mortar production stage. The heterogeneous distribution of some micro-aggregates brought an important problem related with image analysis: the variability may significantly change the values of measured parameters of micro-aggregates depending on their concentration. To overcome this problem the image analysis was based on specific area fraction model. In this model the first operation was the determination of the 2D area fraction of particles on the surface area of each image. The modified version of the proposed method by Wong et al. [25] was used and the critical point of each image was determined by using “cumulative threshold area” method. In that study, the area fraction of pores (dark grey to black regions) was being selected from backscattered electron images. In the case of this study, the target area is the 2D projective surface area of micro-aggregate particles (light grey to white) on secondary electron images captured by SEM. After the determination of 2D area fraction of the particles, second step was the employment of a traditionally used image analysis command (trace contour) to detect the particle shape, angularity and surface morphology differences of each micro-aggregate. Trace contour command as a specific edge contour detection method [26,27] was found to be the best

Table 2
Fineness index (FI) of micro-aggregates

	Quartz fine Qf	Quartz medium Qm	Quartz coarse Qc	River sand fine Rf	River sand medium Rm	River sand coarse Rc	Limestone fine Lf	Limestone medium Lm	Limestone coarse Lc
Fineness index (μm ² /μm ³)	61.33	21.08	12.16	41.71	17.20	12.44	62.80	28.75	18.27

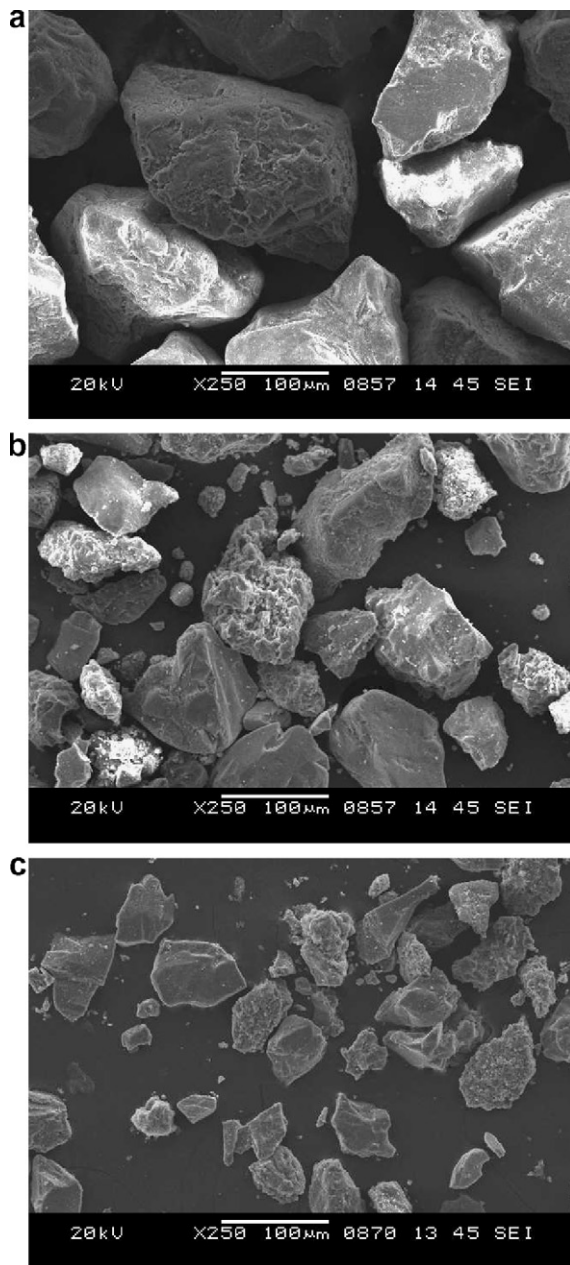


Fig. 3. SEM images of micro-aggregates of quartz sands: (a) Qng, (b) Qc, and (c) Qf.

method to determine the particle characteristics, particularly shape, surface porosity and roughness. Finally the results derived from these analyses were normalised by using the area fractions of each image. These “specific micro-shape and surface texture” values are presented in Fig. 6.

2.3. Mix proportioning, curing, preparation and testing of specimens

Micro-concrete mixtures were designed by targeting the same consistency at the same W/C ratio (0.46). Consistency tests were conducted according to ASTM C187 [28] and flow values were kept constant (125–135 mm) by changing

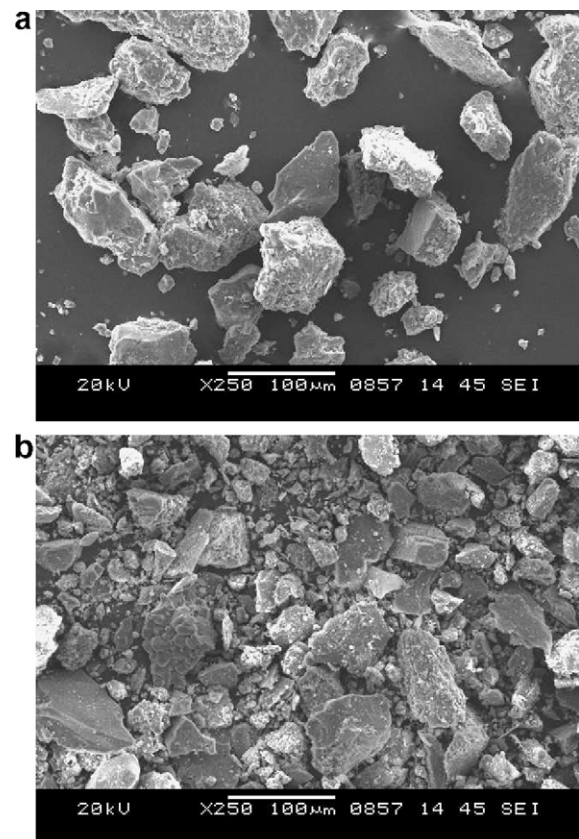


Fig. 4. SEM images of micro-aggregates of river sand: (a) Rc and (b) Rf.

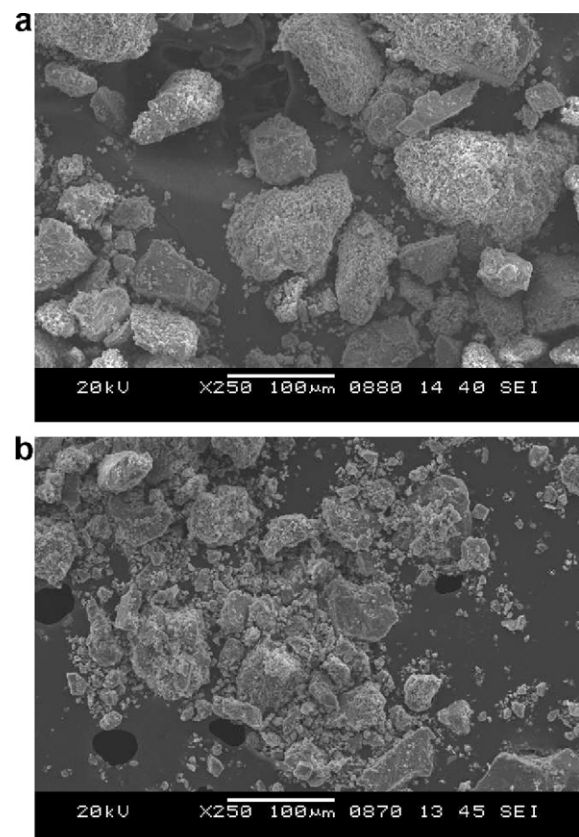


Fig. 5. SEM images of micro-aggregates of limestone: (a) Lc and (b) Lf.

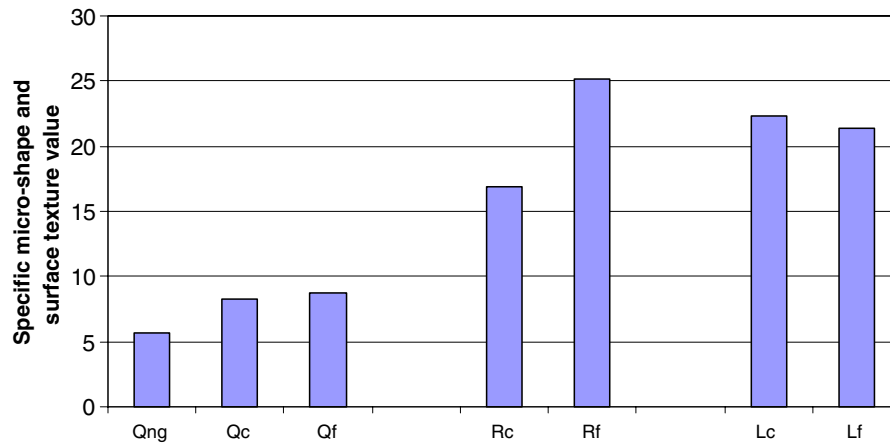


Fig. 6. “Specific micro-shape and surface texture” values of SEM images of micro-aggregates.

Table 3
Mixture proportions of micro-concretes (kg/m³)

Materials	MC-Qc	MC-Qm	MC-Qf	MC-Rc	MC-Rm	MC-Rf	MC-Lc	MC-Lm	MC-Lf
<i>Micro-aggregates</i>									
Portland cement	651	652	658	663	663	661	652	652	652
Silica fume	130	130	132	133	133	132	130	131	130
Water	298	298	301	303	303	302	298	300	298
Fine ground	–	–	1127	–	–	1134	–	–	1117
Medium ground	–	1117	–	–	1137	–	–	1125	–
Coarse ground	1116	–	–	1137	–	–	1118	–	–
Admixture	4.7	7.8	10.5	3.2	7.0	9.5	7.0	10.8	13.3
Total	2200	2204	2228	2239	2242	2240	2205	2218	2210

the admixture dosages. The mix proportioning was based on theoretically filling one cubic meter of volume, like conventional concrete. However, the aggregate phase was reduced due to the extremely high surface area of micro-aggregate particles. At the same time, binder phase (including silica fume) was increased. The aims of using silica fume can be listed as follows: to enhance the particle packing of the system and due to its pozzolanic reactivity to improve the mechanical properties of MC. Pre-trials were conducted to determine the appropriate binder content to maintain the targeted high strength values (>60 MPa). Details of selected MC mix proportions are given in Table 3. The volumetric micro-aggregate content was kept constant for all micro-aggregate types. Slight differences of weights between concrete mixture ingredients were originated from the different specific weights of micro-aggregates. The same mixing sequence was replied for all mixtures. Prismatic samples of 40*40*160 mm were moulded and compacted on a vibration table. A total of five series (including nine samples for each of the batches) were prepared. All samples were moist cured by immersing them in lime-saturated water until the testing ages of 1, 7, 28 days. The flexural and compressive strengths of specimens were determined by using TS EN 196-1 standard [29].

3. Results and discussion

3.1. Effects of FI, micro-shape and surface texture of micro-aggregates on admixture requirement of MCs at constant consistency

FI “a sole number as an indication of PSD change” can be used in the determination of admixture demand of MCs. It is valid that, as the FI increases, the micro-aggregates become finer. The admixture requirements of MCs prepared with micro-aggregates at different finenesses are plotted in Fig. 7. There was a good logarithmic relationship between FI and admixture requirement to obtain constant workability. Note that in cases of limestone and river sand micro-aggregates (Figs. 7b and 7c), the increasing tendency of logarithmic curves by increasing FI was more significant than the quartz originated one (Fig. 7a). The comparison of admixture requirements of MCs at constant fineness index (for example, 40 $\mu\text{m}^2/\mu\text{m}^3$) can be more useful to compare the performance of micro-aggregate types. The admixture requirements of MCs prepared with micro-aggregates of quartz, river sand and limestone at constant FI were 9.1, 9.6 and 11.5 l/m³, respectively.

As a first approximation, FI can be used to calculate the admixture requirement of different types of micro-aggre-

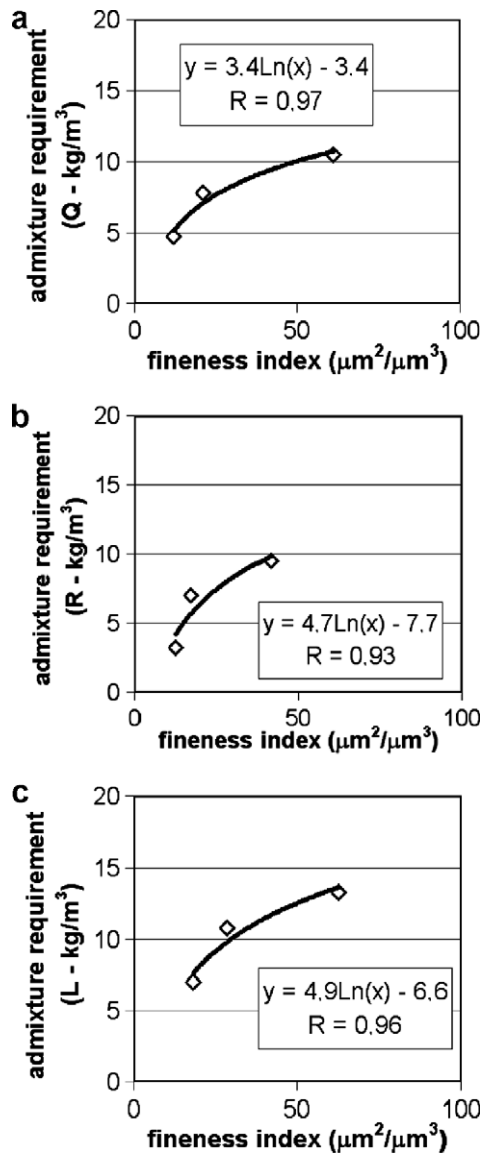


Fig. 7. Relationship between fineness index and admixture requirement for a constant consistency ((a) Q – quartz, (b) R – river sand and (c) L – limestone).

gates incorporated mixes. However, it must be kept in mind that when micro-aggregates from different origins were employed, the correlation may reduce due to the assumption of equivalent sphericity and surface texture differences of micro-aggregates. Micro-aggregates from different sources may have angular surface and needle like or rectangular shape rather than spherical and/or may have rough and porous surface texture. In order to verify this situation, the admixture demands of MC's at constant FI ($40 \mu\text{m}^2/\mu\text{m}^3$) were compared with micro-shape and surface characteristics of micro-aggregates particles. For this purpose the “specific micro-shape and surface texture” values previously calculated by using image analysis methods were plotted against admixture demand (Fig. 8). High correlation coefficients were derived due to the improved reflectance of surface characteristics by using “specific micro-

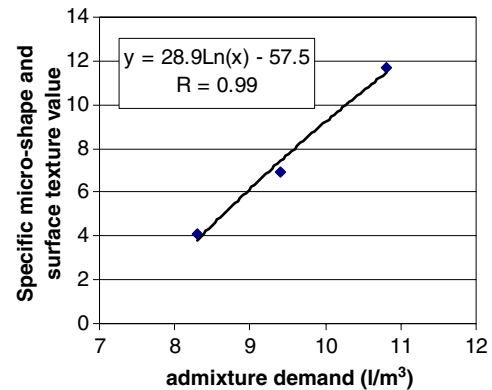


Fig. 8. Relationship between “specific micro-shape and surface texture” value and admixture demand.

shape and surface texture” values. As a result it was proposed that admixture demand is not solely related with the PSD, also particle micro-shape and surface characteristics are important. A better approximation can be obtained by taking the micro-surface characteristics into account.

3.2. Role of high-range water reducing admixture with respect to micro-aggregate characteristics

3.2.1. Surface charge of cement and micro-aggregate particles

Various kinds of forces coexist in cement, micro-aggregates suspension. There are forces of colloidal origin that arise from mutual interactions between particles, and are affected by the polarizability of water. When the van der Waals attraction between cement grains and the electrostatic attraction between unlike charges on the surface of particles are dominant, the net result is an attraction, and the particles tend to flocculate.

In case of micro-aggregates of limestone origin and partially river sand, the increase in fineness index resulted with coagulation of particles even in the absence of water. This situation can be observed from the SEM images of micro-aggregates presented in Figs. 4b and 5b where local coagulation of particles can be identified. If particles also tend to flocculate at dry state, they may also be susceptible to flocculate when contact with water.

However, in the presence of polymeric or surfactant materials on the surface of cement grains and micro-aggregate particles, the net result is repulsion and the particles remain separate. In this respect, the fine ground material can influence the electrostatic forces depending on its mineralogical nature and the state of its particle surface charges. Because colloidal forces depend also on the average distance between nearest neighbour particles, the interposition of finer grains between cement particles may affect their electrostatic attraction and thus their flocculated structure. Note that the effectiveness of admixture also depends on mixing sequence and nature of particle characteristics.

3.2.2. Effects of PSD, shape and surface texture

Broader particle-size ranges were obtained as the FI increased. Theoretically, micro-aggregates with high FI have a higher maximum particle packing possibility because the finer particles may easily fit into the gaps between the coarser particles. Note that, it is not enough to broaden the PSD to obtain maximum particle packing. Other parameters like shape and surface texture should be taken into consideration.

Any deviation from a spherical shape implies an increase in viscosity for the same phase volume. For example, carbonate powder (a precipitated powder) is known to have a shape that is very close to a perfect sphere while the other powders will differ more from the spherical shape and this may affect the rheology. Thus, in the presence of fluidifiers the finer and the more spherical is the filler, the better are the rheological properties. However, the above-mentioned opinions did not take surface texture differences into account. In this study, if Figs. 3–5 are observed, it can be realized that micro-aggregates of quartz have more angular shape when compared to limestone at the similar PSD. However, the surface porosity of micro-aggregates of limestone was extremely higher than that of quartz both at fine and coarse PSDs. From the viewpoint of admixture demand, one can claim that high amounts of admixture should be required for MC prepared with micro-aggregates of quartz due to its angular shape.

On the other hand, high amounts of admixture should also be required for MC prepared with micro-aggregates of limestone due to its high surface porosity. The net result of admixture demand at constant FI showed that the surface texture properties were more dominant in the determination of admixture requirement when micro-aggregates of limestone and quartz were compared. Highly porous surface area increased the admixture absorption capacity of micro-aggregates of fine ground limestone, and the free (effective) admixture content was reduced. In this case, to obtain the same consistency higher admixture content was required.

Fine ground limestone materials can also have negative efficiencies in the adsorption of HRWR admixtures due to their physico-chemical interaction with water. They can introduce soluble calcium ions that may neutralize the chemical activation of HRWR admixtures and affect the kinetics of the hydration reaction, possibly the nucleation of hydration products at initial periods of mixing. All these effects may increase the admixture demand of MC to obtain the targeted consistency. On the other hand, the micro-aggregates of quartz accepted to be inert in the initial periods of mixing.

3.3. Mechanical performance

Test results of the effects of micro-aggregate types and PSD on the compressive strength of MC are presented in Fig. 9. When the micro-aggregates of quartz were focused (Fig. 9a), it was observed that the increase in FI did not

cause any significant compressive strength increase. This can be attributed to the surface texture of micro-aggregates of quartz. As can be observed from SEM images (Fig. 5) while grinding reduced the sizes of the particles, the surface textures were partially roughened. The increase in surface area was not enough to obtain higher compressive strength values. Cracking due to grinding usually results with smaller particles whose surface textures partially preserve their smooth surface structure. On the other hand, the 28 days compressive strength results of all mortars prepared with micro-aggregates of limestone were significantly higher than those prepared with micro-aggregates of quartz (Fig. 9c). If SEM images of limestone-based micro-aggregates are investigated, the higher surface porosity can easily be identified when compared to quartz at the similar PSD (Figs. 3 and 5). The mechanical interlocking capacity between particle and matrix phase, which improves the mechanical performance of transition zone, can directly be related with compressive strength. Poorer mechanical interlocking can be expected in the case of micro-aggregate of quartz.

On the other hand, flexural test results presented in Fig. 10 showed that there is a tendency of decrease in flexural strength when the particle size of micro-aggregates became coarser. Except micro-aggregates of highest fineness index (Qf, Rf and Lf), flexural strength changes with respect to micro-aggregate type were comparatively similar. A significant flexural strength difference was observed between MCs prepared with fine ground micro-aggregates of limestone (Lf) and quartz (Qf). Despite its high compressive strength, the flexural strength of MCs prepared with Lf was lower compared to the MCs prepared with Qf. The unexpected low flexural strength of MCs prepared with Lf can be attributed to the random particle coagulation susceptibility of limestone particles. As mentioned before limestone particles (if finely ground) had a tendency to coagulate (Fig. 5). Note that the local coagulation of these particles may entrap very small capillary pores if the viscosity of matrix phase is high or inadequate mixing was applied. This may increase the local micro-entrapped air content of matrix phase, which results with a more heterogeneous concrete network. In case of application of compressive stress, these micro-pores will tend to close with time due to the increased compressive strain and will have no negative influence on compressive strength. On the other hand, the mode of collapse is extremely different in case of flexure stress. The randomly distributed micro-porosity accumulations as the weakest chains of the link at cross-section may play a significant role in the determination of flexural strength. Note that in case of micro-aggregates of quartz, there is no evidence of coagulation (Fig. 3). As a result more homogeneously dispersed micro-pore system can be obtained. In this study, the mixing time was kept constant for all mixtures. It seems that this time might not be enough to disturb the local coagulation of limestone particles even in the presence of higher amounts of HRWR admixture. As a general statement it can be

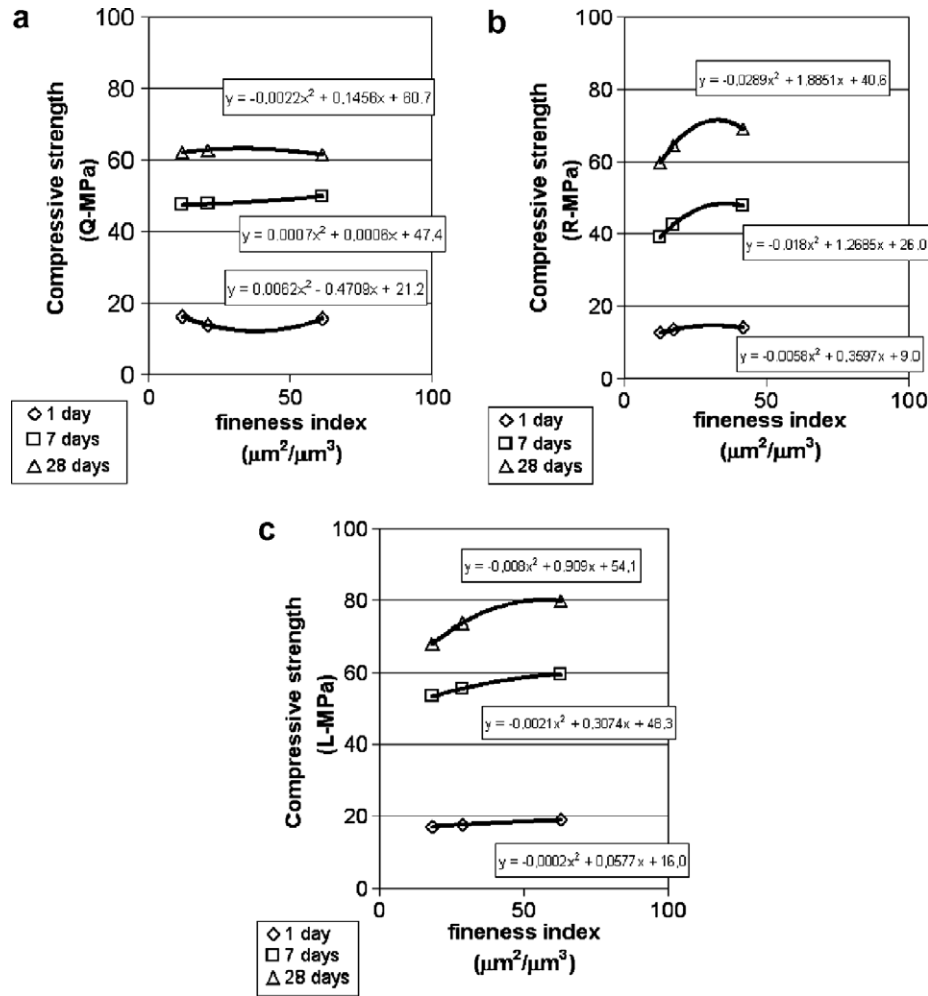


Fig. 9. Relationship between fineness index and compressive strength of MC mixes.

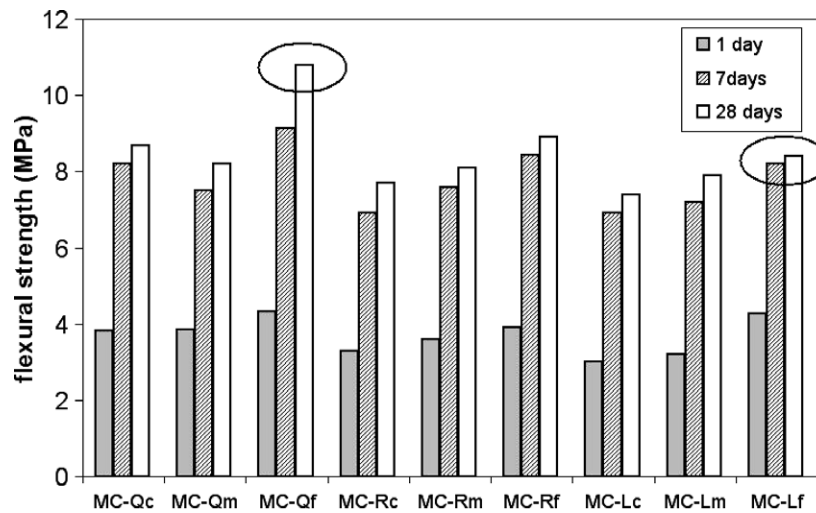


Fig. 10. Flexural strengths of MC specimens.

interpreted that mixing time should be increased to guarantee the homogeneous dispersion of fine ground micro-

aggregates of limestone in order to obtain higher flexural strength values.

4. Conclusions

The first step of mixture proportioning of MC is the characterization of micro-aggregates. In addition to SEM images supported by digital analysis methods, the concept of “Fineness Index” containing both surface area and volume parameters in itself can be used for the characterization of micro-aggregates. The comparison of different micro-aggregates can give valuable information to designers for both in determination of fresh properties (admixture demand to obtain constant consistency) and in predicting mechanical performance. The main conclusions derived from this study are divided into two parts:

1. The surface porosity of micro-aggregates of limestone was extremely higher than quartz both at fine and coarse PSDs. The net result of admixture demand comparisons at constant FI showed that the surface texture properties were more dominant in determination of admixture requirement when micro-aggregates of limestone and quartz were compared.
2. In the case of extremely ground limestone and quartz (Lf and Qf), the effect of micro-aggregate type on compressive strength was not parallel to the effect of flexural strength. This can be attributed to the possible coagulation of fine ground limestone due to the different surface roughness compared to quartz in fresh MC system.

References

- [1] Mehta PK. Advancements in concrete technology. *Concr Int* 1999; 21(6):69–76.
- [2] Long G, Wang X, Xie Y. Very-high-performance concrete with ultra fine micro fillers. *Cement Concrete Res* 2001;32(4):601–5.
- [3] Nehdi M, Mindess S, Aitcin P-C. Rheology of high-performance concrete: effect of ultra fine particles. *Cement Concrete Res* 1998; 28(5):687–97.
- [4] Aitcin P-C. Developments in the application of high-performance concretes. *Constr Build Mater* 1995;9(1):13–7.
- [5] Wallevik OH, Saasen A, Gjorv OE. Effect of filler materials on the rheological properties of fresh concrete. *ACI Mater J* 1995;92(5): 524–8.
- [6] Herrmann HJ, Baram RM, Wackenhut M. Searching for the perfect packing. *Physica A* 2003;330(1–2):77–82.
- [7] Saak AW. Characterization and modelling of the rheology of cement paste: with applications toward self-flowing materials. Northwestern University, PhD Thesis; 2000. p. 283.
- [8] Ferraris CF, de-Larrard F. Testing and modelling fresh concrete rheology. National institute of standards and technology. NISTIR, Maryland, Report No. 6094; 1998. p. 71.
- [9] Mindess S, Young JF, Darwin D. *Concrete*. 2nd ed. NJ: Prentice Hall; 2002, ISBN 0130646326. p. 644.
- [10] Funk JE, Dinger DR. Particle size control for high solids castable refractories. *Am Ceram Soc Bull* 1994;73(10):66–9.
- [11] Stovall T, de-Larrard F, Bui M. Linear packing density model of particulate mixtures. *Powder Technol* 1986;48(1):1–12.
- [12] de-Larrard F, Sedran T. Optimization of ultra-high performance concrete by the use of a packing model. *Cement Concrete Res* 1994;24(6):997–1009.
- [13] Dinger DR, Funk JE. Particle-packing phenomena and their application in materials processing. *MRS Bull.* 1997;22(12):19–23.
- [14] Williams DA, Saak AW, Jennings HM. The influence of mixing on the rheology of fresh cement paste. *Cement Concrete Res* 1999;29(9): 1491–6.
- [15] Bentz DP, Garboczi EJ, Snyder KA. Hard core/soft shell micro structural model for studying percolation and transport in three-dimensional composite media. National institute of standards and technology. NISTIR, Maryland, Report No. 6265; 1999. p. 55.
- [16] Sedran T, de-Larrard F. RENE-LCPC: software to optimize the mix-design of high-performance concrete. In: de Larrard F, Lacroix R, editors. *Proceedings of the fourth international symposium on the utilization of high-strength/high-performance concrete*; 1996. p. 169–78.
- [17] Chang C, Powell RL. Effect of particle size distributions on the rheology of concentrated bimodal suspensions. *J Rheol* 1994;38(1): 85–98.
- [18] de Larrard F. *Concrete mixtures proportioning a scientific approach*. Modern concrete technology series. Routledge: E&FN SPON; 1999, ISBN 0419235000. p. 421.
- [19] Powers TC. *The properties of fresh concrete*. John Wiley and Sons; 1968, ISBN 0471695904. p. 664.
- [20] Pedersen B, Mortsell E. Characterization of fillers for SCC. In: Ozawa K, Ouchi M, editors. *Proceedings of the second international symposium on self-compacting concrete*, RILEM S.A.R.L., Tokyo, Japan; 2001. p. 257–66.
- [21] ASTM C494-99a. Standard specification for chemical admixtures for concrete. *Annual Book of ASTM Standards*; 2002.
- [22] Orhan M, Özer M, Işık NS. Investigation of laser diffraction and sedimentation methods which are used for determination of grain size distribution of fine grained soils. *GU J Sci* 2004;17(2):105–13.
- [23] Ferraris CF, Hackley VA., Aviles AI, Buchanan CE. Analysis of the ASTM round-robin test on particle size distribution of Portland Cement: Phase I. National institute of standards and technology. NISTIR, Maryland, Report No. 6883; 2002. p. 51.
- [24] Hackley VA, Lum L-S, Gintautas V, Ferraris CF. Particle size analysis by laser diffraction spectrometry: application to cementitious powders. National institute of standards and technology. NISTIR, Maryland, Report No. 7097; 2004. p. 66.
- [25] Wong HS, Head MK, Buenfeld NR. Pore segmentation of cement-based materials from backscattered electron images. *Cement Concrete Res* 2006;36(6):1083–90.
- [26] Achour K, Zenati N, Laga H. Contribution to image and contours restoration. *Real-Time Imaging* 2001;7(4):315–26.
- [27] Chen T, Wu QH, Torkaman RR, Hughes J. A pseudo top-hat mathematical morphological approach to edge detection in dark regions. *Pattern Recogn* 2002;35(1):199–210.
- [28] ASTM C 187. Standard test method for normal consistency of hydraulic cement. *Annual book of ASTM standards*; 2002. p. 2.
- [29] TS EN 196-1. *Methods of testing cement: determination of strength*. Turkish Standards, TSE, Ankara; 1994. p. 19.