REVISTAMATÉRIA
ISSN 1517-7076 artigos e13227, 2022

V.27 N.02

Influence of particle size of aggregates on the rheological behavior of crowded suspensions with silicone oils with different viscosities

Influência do tamanho das partículas dos agregados no comportamento reológico de suspensões concentradas com óleos de silicone de diferentes viscosidades

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RESUMO

Dois tamanhos diferentes de agregados de microesferas de vidro foram comparados em suspensão com óleos de silicone de diferentes viscosidades. A concentração da suspensão foi mantida constante e igual a 62% vv. O comportamento reológico das suspensões foi avaliado pelo teste de *squeeze-flow*, para duas velocidades de ensaio. Para ambos os tamanhos de partícula, quando imersas no óleo de silicone menos viscoso, a tensão de escoamento não apresentou boa correlação com as velocidades de ensaio. Enquanto isso, o óleo de silicone mais viscoso levou a uma boa correlação entre a tensão de escoamento e as velocidades de ensaio. Para granulometria mais fina, a "viscosidade" das suspensões aumenta com o aumento da viscosidade dos óleos de silicone e com a velocidades de ensaio. Para a granulometria mais grossa, a "viscosidade" das suspensões aumenta com as velocidades de ensaio e diminui conforme a viscosidade dos óleos de silicone aumenta. Essas diferenças no comportamento reológico estão intimamente relacionadas à ocorrência de fluxo não homogêneo, que depende da relação entre a velocidade de percolação dos óleos de silicone e as velocidades de ensaio do teste de *squeeze-flow*. Assim, o tamanho dos agregados influencia de diferentes maneiras o comportamento reológico dessas suspensões de microesferas de vidro. Consequentemente, a combinação da viscosidade da fase da matriz com a granulometria dos agregados pode levar a um escoamento homogêneo.

Palavras-chave: micro esferas de vidro, óleo de silicone, *squeeze-flow* e percolação.

ABSTRACT

Two different sizes of aggregates of glass microspheres were compared in suspension with silicone oils of different viscosities. The concentration of the suspension was kept constant and equal to 62 vv.%. The rheological behavior of the suspensions was evaluated by the squeeze flow test, for two test speeds. For both particles size, when immersed in less viscous silicone oil, the yield stress did not show a good correlation with the tests speeds. Meanwhile, the more viscous silicone oil led to a good correlation between the yield stress and tests speeds. For the finer granulometry, the "viscosity" of suspensions increases with increasing viscosity of silicone oils and test speeds. But for the coarser granulometry, the "viscosity" of suspensions increases with the test speeds, and decreases as the viscosity of the silicone oils increases. These differences in the rheological behavior are closely related to the occurrence of non-homogenous flow, which depends on the relation between the percolation speed of silicone oils and the test speeds of the squeeze flow test. Thus, the size of particles influences in different ways the rheological behavior of these suspensions of glass microspheres. Consequently, a combination of the matrix phase viscosity and granulometry of aggregates phase can lead to a homogeneous flow.

Autor Responsável: Thiago Melanda Mendes Data de envio: 22/01/2021 Data de aceite: 24/01/2022

10.1590/S1517-707620220002.1327

Keywords: glass microspheres, squeeze flow, silicone oils, percolation.

1. INTRODUCTION

The squeeze flow test [1-4], was successfully applied for the characterization of cement pastes [5] and mortars [6-9], due to its ability to simulate application of these suspensions as a rendering mortars or concrete tiles, which are subjected to a thickness variation imposed by a plastic conformation. In addition to providing fundamental rheological parameters of the suspension, such as viscosity and yield stress [5-9], the squeeze flow test was also able to identify the occurrence of phase separation (bleeding or segregation) also called non-homogeneous flow [10, 11]. Conceptually, the pastes, mortars and concrete can be considered as multiphase suspensions, the first represents a dispersion of particles (< 75µm) in an aqueous media, the mortar can be defined as a dispersion of sand in a cementitious paste, and for concrete, the gravel or coarse aggregates are dispersed in the mortar [12-14]. In this way, particles coarser than 75 µm such as the sand or gravel, can be considered as aggregates. The phase separation, observed during the bleeding or segregation, has a direct effect on the homogeneity and on the quality of the final product of the cement-based materials. This heterogeneous flow occurs mainly when the suspensions are under very low velocities, close to zero, or casted in a mold, for example. The viscosity of the "fluid" phase, and the permeability of the "solid" phase must also be considered in the heterogeneous flow observed in Portland cement-based materials. Recently, Mendes *et al*. 2020 [5] evaluated the rheological behaviour of cement pastes and mortars applying the squeeze-flow tests. For cement pastes and mortars formulated with a fixed granulometry of glass microspheres, the yield stress and viscosity of these cement-based materials were closely related with interparticle separation (IPS) of cement paste. This complex multiphase nature of cement paste and mortars can lead to a misunderstanding of the effect of each variable on the rheological behaviour of cement-based materials. Since the particle-to-particle interactions between cement and aggregates was not considered, as well the reaction time dependence of reactive particles could not be isolated. In this way, the study of a rheological model, composed by a fluid without particles in suspension for acts as a paste or a matrix, is another approach to understand this problem. Similarly, the aggregates used in mortars present many characteristics, such as chemical composition and morphology, which can be isolated by the use of glass microspheres. Those has the same chemical nature of silicone oils, avoiding any type of physical or chemical interaction between these phases.

NIKKHOO and GADALA-MARIA [15] evaluated the effect of liquid viscosity, particle size and solids concentration of suspensions of fine microspheres (50-100 µm) under squeeze flow for a constant force. The results indicate, however, that liquid-phase migration increases with increasing initial solid volume fraction, increase of the particle size, and decrease of the viscosity of the liquid. The experimental data from the referred paper suggested that the migration of the liquid phase can be avoided, by increasing the squeeze speed, increasing the viscosity of the liquid, using smaller particles, or decreasing the initial volume fraction. CHEN, OYE AND SJOBLOM [16] evaluated glass microspheres of fine particles (90 µm) dispersed in mineral oil (γ = 0.85 g/cm3 and viscosity of 0.12 Pa.s). Authors showed a shear thickening behavior for suspensions of 30% in volume of these glass microspheres. According to them this behavior is closely related to the sedimentation effects of these particles. DELHAYE, POITOU AND CHAOUCHE [10] and COLOUMB, CHAARI AND CHAOUCHE [11], and analyzed fine particles (<100 µm) immersed in liquids with different viscosities. They demonstrated that these suspensions behave homogeneously when the speed imposed on the suspension is equal the percolation speed of the matrix, and that if the matrix moves faster than the speed imposed on the suspension, a heterogeneous flow occurs between the phases. For aggregates with particles larger than 1000 µm, MENDES [17] and SAKANO *et al*. [18] evaluated the influence of squeeze flow speed and silicone oil viscosity on the rheological behavior of crowded suspensions. For a unique particle size of glass microspheres, they suggested that the phases separation or non-homogeneous flow is related to the permeability, viscosity of silicone oils and squeeze flow speeds evaluated. In this way, considering that the permeability of the aggregates depends on their particle size. There is a lack in the knowledge about the influence of the aggregate size (> 75 µm) on the rheological behavior of crowded suspensions. This study aims to evaluate the influence of the particle size of aggregates on the rheological behavior of crowded suspensions.

2. MATERIALS AND METHODS

Two different sizes of glass microspheres aggregates were obtained by sieving. The intervals between 1000 and 850 µm, and 300 and 250 µm were selected. An average value was adopted to represent these particles sizes, 925 and 275 µm, respectively. Figure 1 shows the micrographies of the glass microspheres. Table 1 shows their physical characteristics. The apparent density was measured according to [19] and real density was obtained from gas picnometry according to [20]. Factorial design was used in order to stablish a statistical approach for the rheological tests. Three repetitions were performed for each test configuration, allowing the selection of two variables for the comparison analysis, the squeeze flow speed (X_1) and the viscosity of silicone oils (X_2) [21]. The values and signs of these variables are listed in Table 2. Figure 2 shows the configuration, squeeze flow tests the samples were kept in a plastic ring with a diameter of 100 mm and a height of 20 mm, using an upper piston with a diameter of 25.4. The samples were deformed to 10 mm, resulting in a final height of 10 mm. All measurements were performed on an Instron 5569 universal machine using a 1 kN load cell. After each test, the samples were removed from plastic ring, mixed in a bowl and measured again. The solids concentration of suspensions was kept constant and equal to 62 %.vv. Consequently, the volume of silicone oils was kept constant and equal to the volume of voids between the aggregates. These values were calculated from the apparent and real densities of the aggregates fractions (Table 2).

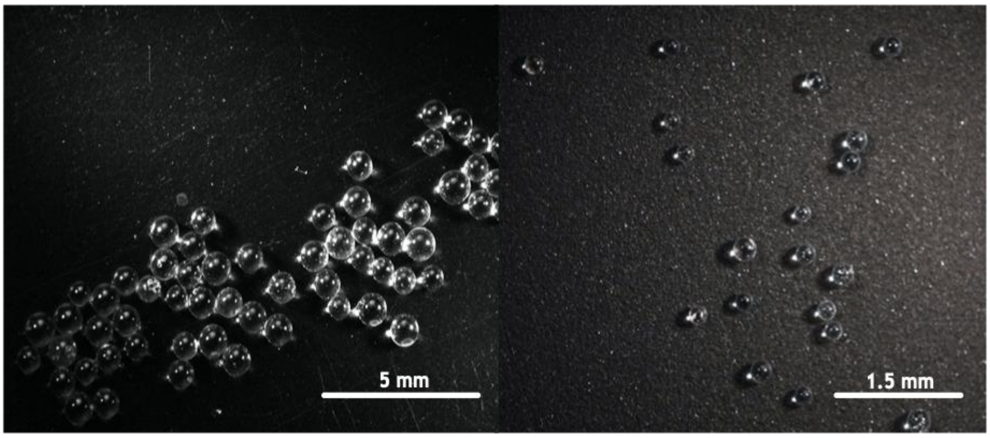

Figure 1: Glass microspheres (a) 1000-850 μm (b) 300-250 μm

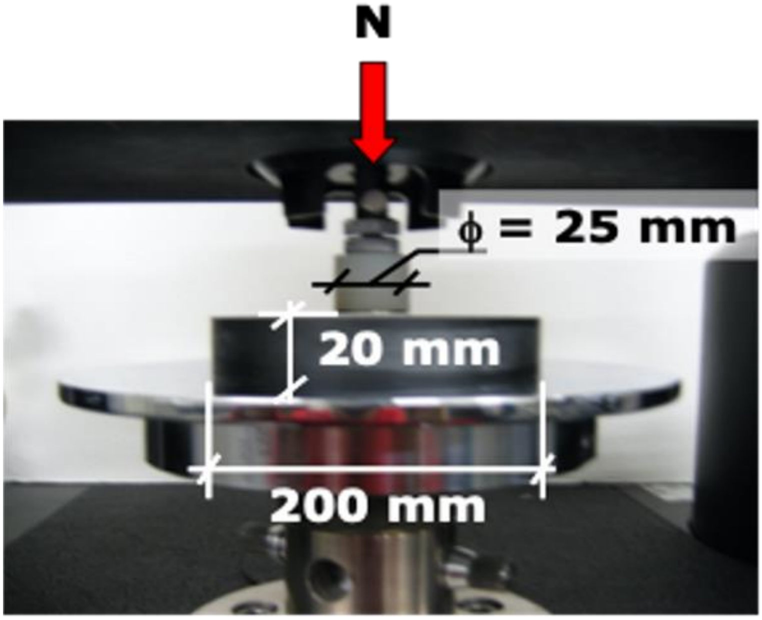

Figure 2: Test configuration of squeeze-flow

Table 1: Physical characteristics of glass microspheres

1000 – 850 μm	**300 – 250** μm
Real density (g/cm^3)	Real density (g/cm^3)
2,501	2,493
Apparent density (g/cm^3)	Apparent density (g/cm^3)
1,543	1,547

Table 2: Variables considered for Factorial design

X_1 – SILICONE OIL VISCOSITY	X_2 – SQUEEZE FLOW SPEED
10^3 cSt (-)	0.1 mm/s (-)
10^3 cSt (-)	1.0 mm/s (+)
10^4 cSt (+)	0.1 mm/s (-)
10^4 cSt (+)	1.0 mm/s (+)

3. RESULTS

Figure 3 shows the stress x displacement curve for the suspensions of 300-250 µm (a) and 1000 – 850 µm (b) silicone oils of 1000 cSt, and squeeze flow speeds of 0.10 mm/s. A linear adjustment was used to calculate the elasticity and yield stress of these suspensions, the inclination of tangent and the constant values for the linear equation y = a*x + b, respectively.

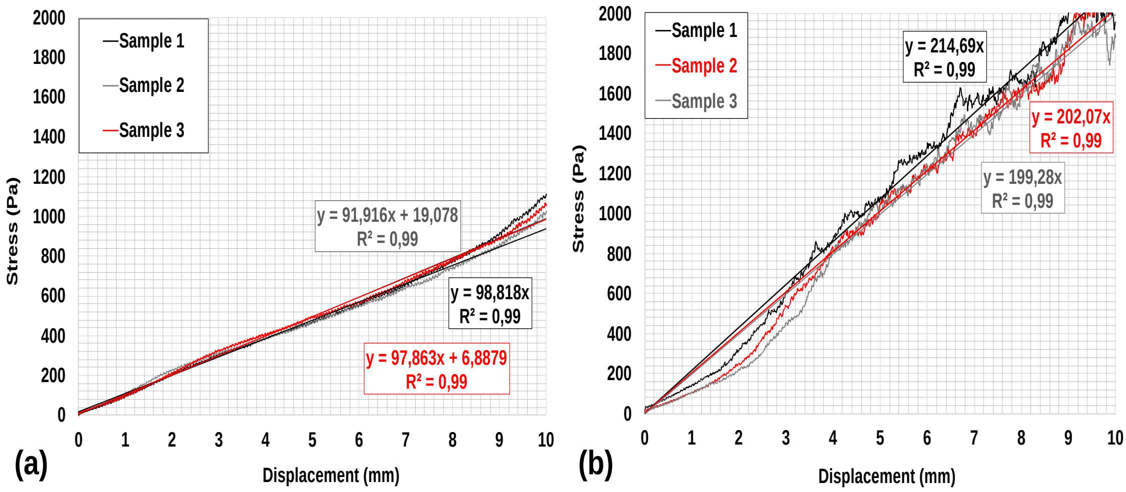

Figure 3: Squeeze-flow tests (a) 300-250 µm + 1000 cSt – v = 0.1 mm/s (b) 1000-850 µm + 1000 cSt – v = 0.1 mm/s

Table 3 presents the calculated values of the yield stress and elasticity of crowded suspensions with particles 300-250 µm for the studied conditions. Figure 4 (a) shows the influence of silicone oil viscosity (X_1) and squeeze flow speed (X_2) on the yield stress of suspension 300 – 250 µm. As seen, in the suspension with the most viscous silicone oil (10000 cSt), the yield stress increases according to the squeeze flow speeds, and a good correlation (R^2 = 0.78) was observed. For a stationary condition (v = 0 mm/s), the yield stress can be estimated at 67 Pa. But, when the less viscous silicone oil (1000 cSt) was considered, the yield stress does not show a good correlation (R^2 = 0.30) with the squeeze flow speeds. For the stationary condition, a minimum value was estimated at 5 Pa. For this particle size, 300-250 µm, the highest yield stress values were observed for the faster squeeze speed (1 mm/s) and the silicone oil more viscous (10000 cSt). And the squeeze flow speed has a greater influence than the viscosity of silicone oils for this property. Applying a Fisher test to compare the variables, only the test conditions for 0.1 mm/s and 10000 cSt and 1 mm/s and 1000 cSt can be considered statically equal. Figure 4 (b) shows the influence of viscosity of silicone oils (X_1) and squeeze flow speed (X_2) on the elasticity of suspension 300 – 250 µm. A good correlation between the elasticity of crowded suspension and squeeze flow speeds was achieved for both silicone oils, R^2 = 0.98 and R^2 = 0.97, for 10000 cSt and 1000 cSt, respectively. The elasticity of crowded suspensions increases as the squeeze flow speeds and the viscosity of the silicone oils increase. Higher values were observed for faster squeeze flow speeds and more viscous silicone oils. The statistical comparison of the values by the Fischer test, indicated that the conditions of tests of 1 mm/s and 1000 cSt and 0.1 mm/s and 10000 cSt showed no significant difference for the elasticity of these crowded suspensions. Likewise, for the yield stress, the elasticity of crowded suspensions is more influenced by the squeeze flow speed than by the viscosity of silicone oils. For suspensions of polymethylmethacrylate (PMMA) particles with sizes between 300 and 250 μm immersed in 1000 cSt silicone oil, of NIKKHOO *et al.* [22, 23] published values for the yield stress varying from 71 to 132 Pa, for solids concentrations of 0.54 and 0.62, respectively. These values are in the same order of magnitude as those obtained in this research, but the controlled force condition employed by them can explain the difference in absolute values.

Table 3: Results from squeeze flow of micro spheres 300 – 250 μm and 1000 and 10000 cSt for velocities of 0.1 and 1.0 mm/s

D_P (mm)	SPEED (mm/s)	VISCOSITY (cSt)	YIELD STRESS OF SUSPENSION (Pa)	ELASTICITY OF SUSPENSION (Pa/mm)
275	0,1	1000	0	98,82
275	0,1	1000	6,88	97,86
275	0,1	1000	19,07	91,91
275	1	1000	0	151,38
275	1	1000	81,47	162,01
275	1	1000	40,28	166,58
275	0,1	10000	106,71	139,38
275	0,1	10000	175,05	116,8
275	0,1	10000	57,21	135,84
275	1	10000	697,25	239,56
275	1	10000	508,75	237,44
275	1	10000	350,97	243,35

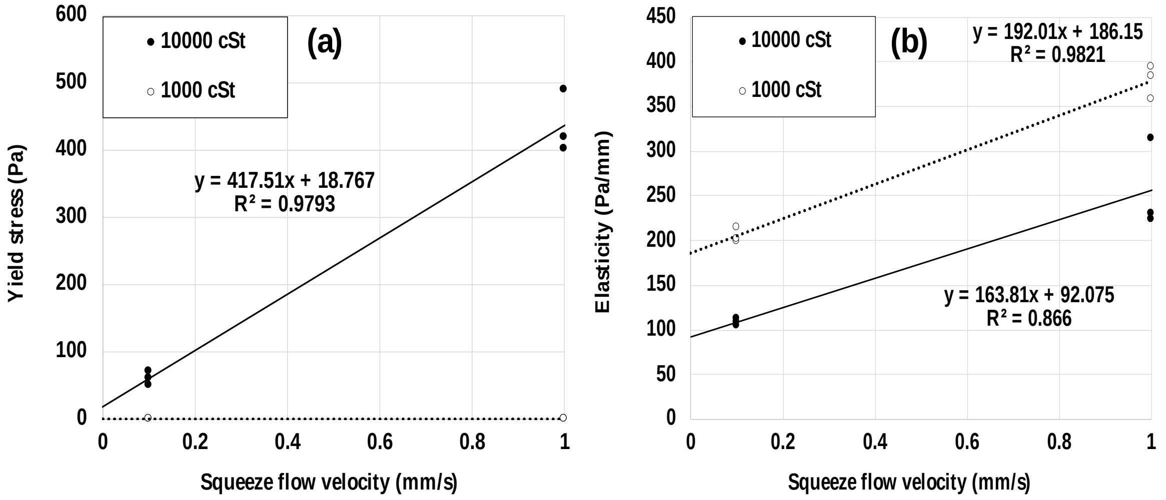

Figure 4: Influence of viscosity of silicone oil (X_1) and squeeze flow velocity (X_2) on the yield stress (a) and elasticity (b) of crowded suspension 300-250 μm

Figure 5 (a) shows the influence of viscosity of silicone oils (X_1) and squeeze flow speed (X_2) on the yield stress of suspension 1000 – 850 μm. For less viscous silicone oil (1000 cSt) the yield stress is zero for both speeds. For the more viscous silicone oil (10000 cSt) the yield stress increases with the increase of squeeze flow speed, showing a good correlation between them (R^2 = 0.97). Considering the Fisher's comparisons, only the yield stress for 1000 cSt silicone oils and squeeze flow speeds of 0.1 and 1 mm/s can be considered equal. Figure 5 (b) shows the influence of silicone oil viscosity (X_1) and squeeze flow speed (X_2) on the elasticity of crowded suspensions with particles 1000-850 µm. For both 1000 and 10000 cSt silicone oils, the elasticity of the suspensions increases when the squeeze flow speed increase. However, unlike crowded suspensions with particles 300-250 µm, the "elasticity for less viscous silicone oil (1000 cSt) is greater than the values for the silicone oil with 10000 cSt. These results indicate that only the combination of the coarse particle size (925 µm) and the most viscous silicone oil (10000 cSt), reached a homogeneous flow. Allowing the best lubrication between the particles, reducing the energy dissipation of particles as friction. Applying the Fisher's test to statically compare the results of the elasticities of these crowded suspensions, all combinations can be considered different. SAKANO *et al.* [11] published values of 0 Pa for the yield stress of crowded suspensions of particles 1180-1000 µm and 1000 cSt silicone oil for 0.1 and 1 mm/s squeeze flow speeds. However, for the crowded suspensions of this aggregates size, immersed in 10000 cSt silicone oil, the authors presented values of 1685 Pa and 178 Pa for the squeeze flow speeds of 1 and 0.1 mm/s, respectively. Taking into account the elasticities, the results published by them reached values around 831 and 286 N/mm for the silicone oils with 10000 cSt, and speeds of 1 and 0.1 mm/s. For silicone oil with viscosity of 1000 cSt the published results were about 781 and 392 N/mm, for squeeze flow speeds of 1 and 0.1 mm/s, respectively. The wall effect due to the ratio between particle size and sample height may explain this different behavior from the values obtained in the present study. For an initial height of the sample (20 mm) and the average size of evaluated glass microspheres (1.080 µm), a ratio of 20/1.08 = 18.51 was evaluated by [18]. Which implies a values lower than 20, and the most probably occurrence of the wall effect for their sample configuration. For the present study, this ratio was 20/0.925 = 21.82, which reduced the possibility of the wall effect for the sample configuration. The numbers of repetitions evaluated is another possible reason for this difference. A single measurement reported in the present study [18].

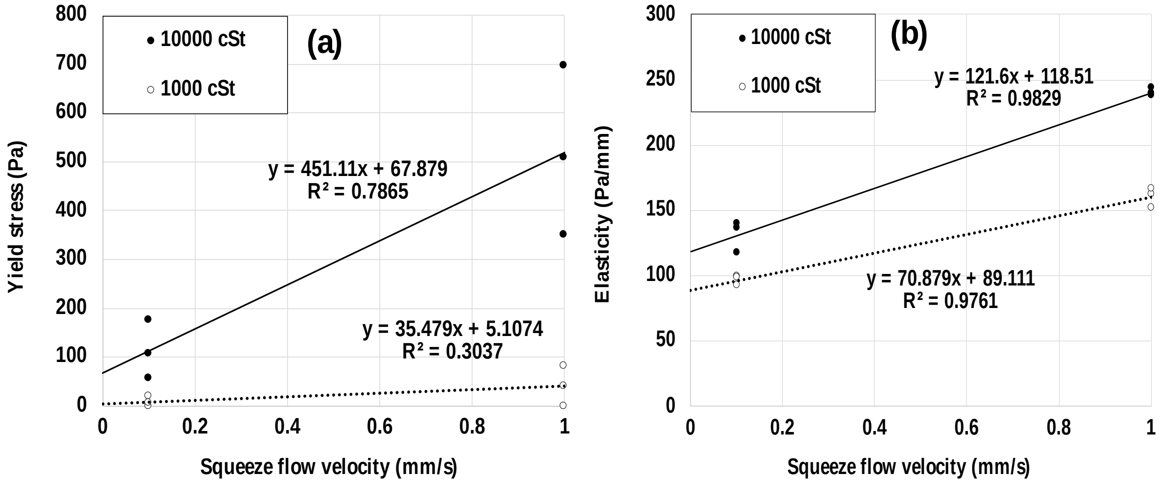

Figure 5: Influence of viscosity of silicone oil (X_1) and squeeze flow velocity (X_2) on the yield stress (a) and elasticity (b) of crowded suspension 1000-850 µm

Table 4: Results from squeeze flow of micro spheres 1000 – 850 µm and 1000 and 10000 cSt for velocities of 0.1 and 1.0 mm/s

D_P (mm)	SPEED (mm/s)	VISCOSITY (cSt)	YIELD STRESS OF SUSPENSION (Pa)	ELASTICITY OF SUSPENSION (Pa/mm)
925	0,1	1000	0	214,69
925	0,1	1000	0	199,28
925	0,1	1000	0	202,07
925	1	1000	0	393,33

925	1	1000	0	383,53
925	1	1000	0	357,62
925	0,1	10000	59,797	112,64
925	0,1	10000	50,313	108,41
925	0,1	10000	71,444	104,32
925	1	10000	418,61	313,54
925	1	10000	488,63	230,48
925	1	10000	401,58	223,65

4. DISCUSSION

These different particle size influences on the rheological behavior of crowded suspensions with 300-250 and 1000-850 μm is directly related to the occurrence of a non-homogeneous flow. NIKKHOO *et al.* [23] and HOYLE *et al.* [24] attributed the non-homogeneous flow, or the fluid migration of very fine glass microspheres to the sedimentation phenomenon. For the condition evaluated here, the glass microspheres were not "suspended" in liquid medium. The volume of voids was filled with the silicone oils. The "filtration" process or even a percolation phenomenon was the most probably situation involved in the phase separation. Considering that the velocity of percolation of a fluid in a granular medium can be estimated by Darcy's law, according to the Equation 1.

$$V_L = k * \frac{\Delta P}{L} = P_0 * \frac{L}{L_E} * \frac{{P_0}^2}{(1 - P_0)} * \frac{{V_S}^2}{2\eta S^2} * \frac{\Delta P}{L} \qquad (1)$$

where:

V_L – is the velocity of percolation of a fluid (mm/s);

P_0 – is the porosity of the granular medium (%);

L_E – is the equivalent length of the sample (mm);

V_S – is the solid concentration of sample (%);

η – is the viscosity of the fluid (Pa.s);

S – is the contact area between the fluid and the granular medium (mm^2);

D_P – is the pression difference applied in the fluid (Pa);

L – is the thickness in the flow direction (mm);

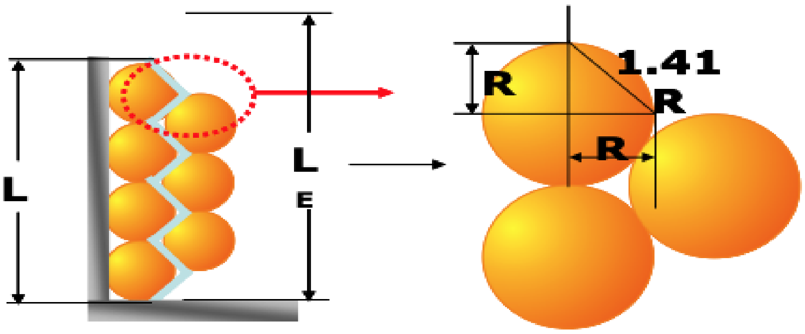

Figure 6: Schematic representation of equivalent length for monodispersions of spheres [25].

Applying the Equation 1, the velocities of the percolation were calculated for the silicone oils with viscosities of 1000 and 10000 cSt, 1 and 10 Pa.s, respectively, for the 275 and 925 μm microspheres. The contact area between the fluid and the particles (S) was estimated from the area of the spheres present in 1 cm^3 of the sample. The porosity was assumed as 38%.vv. and solids concentration as 62%.vv. The length (L) and equivalent length (L_E) and of sample were assumed as 1 and 1.41 L, respectively. And the pressure gradient ($\Delta P/L$) was assumed to be 1. Figure 6 shows the schematic representation of the equivalent length (L_E) of a monodispersion of spheres [25].

Table 05: Permeability properties of glass micro-spheres and percolation velocity of silicone oils

D_P (mm)	SILICONE OIL VISCOSITY (Pa.s)	CONTACT AREA BETWEEN FLUID AND SOLIDS (m^2)	VELOCITY OF LIQUID PERCOLATION (m/s)
925	1.0	$2.59*10^{-3}$	2023
925	10	$2.59*10^{-3}$	202
275	1.0	$8.72*10^{-3}$	178
275	10	$8.72*10^{-3}$	17.8

Figure 7 shows the values estimated for the percolation velocities of a fluid in the glass microspheres (Table 5), the squeeze flow speeds, and the yield stress of the crowded suspensions. As seen, the yield stress of the crowded suspensions did not reach a good correlation with these two variables ($R^2 = 0.44$). Figure 8 shows the influence of the percolation velocity of a liquid (V_L) and squeeze flow speeds (V_S) on the "viscosity" of the crowded suspensions. For this property these variables showed a good correlation with these two velocities ($R^2 = 0.86$), the values increase as both speeds increase. In this way, the low percolation velocity leads to a homogeneous flow, even for large particles, when combined with very viscous silicone oils. When the percolation velocity increases, phase separation occurs, allowing the predominance of friction events between the coarse particles.

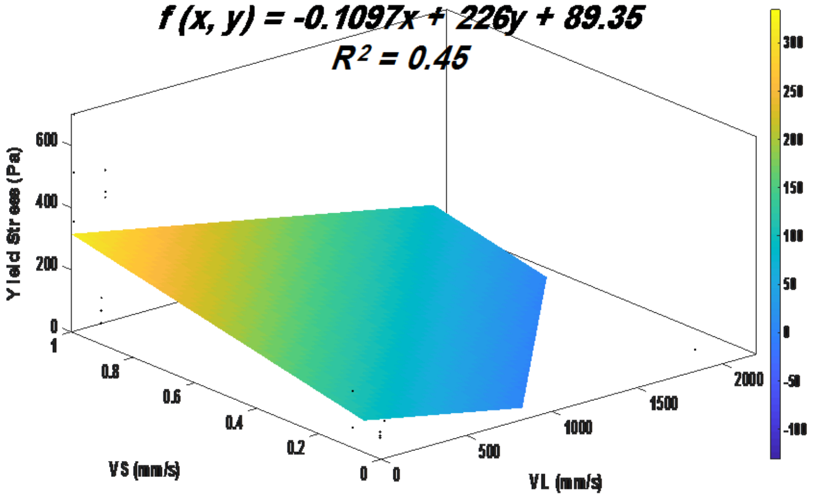

Figure 7: Influence of velocity of liquid percolation (V_L) and squeeze-flow speeds (V_S) on the yield stress of crowded suspensions (Pa)

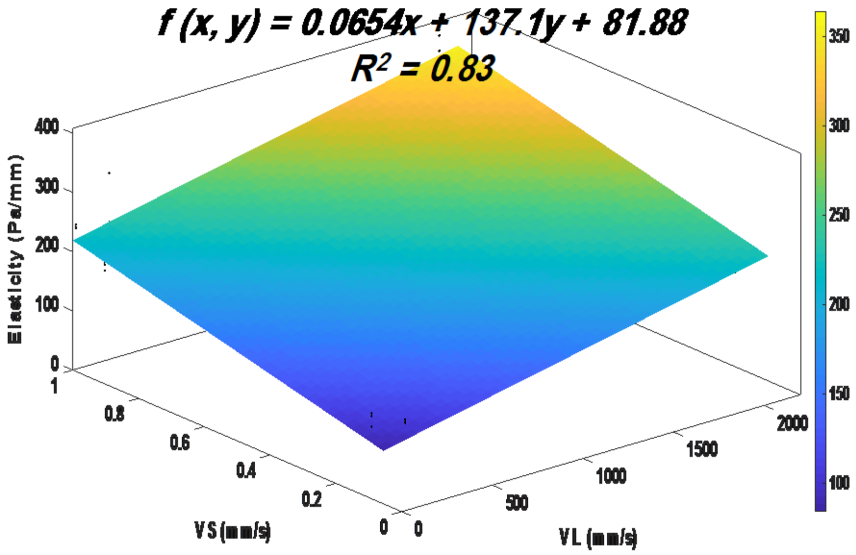

Figure 8: Influence of velocity of liquid percolation (V_L) and squeeze flow velocities (V_S) on the “viscosity” of crowded suspensions (Pa)

5. CONCLUSIONS

The particle size of aggregates influences the rheological behavior of crowded suspensions in different ways:

- For the finer granulometry (300-250 μm), when combined with the more viscous silicone oil, the yield stress and “viscosity” of crowded suspensions showed a good correlation with the squeeze flow speeds. When the less viscous silicone oil was considered, the yield stress did not show a good correlation with squeeze flow speeds, but “viscosity” of crowded suspensions increases according to the squeeze flow speeds. If the viscosity of silicone oil increases, the viscosity of crowded suspensions increases;

- For the larger granulometry (1000-850 μm), when combined with the more viscous silicone oil, the yield stress and “viscosity” of crowded suspension show a good correlation with the squeeze flow speeds. When the less viscous silicone oil, was considered, the yield stress was null, and the “viscosity” increases according to the squeeze flow speeds. If the viscosity of silicone oil increases, the viscosity of crowded suspensions decreases;

- When combined with viscous silicone, the coarse granulometry presents a percolation velocity of the liquid phase similar to the finer particles. However, when the percolation velocity of the matrix increases, the suspension does not present a homogenous flow, and the “viscosity” of crowded suspensions increases. If the lubricant capacity of the matrix phase is low due to the phase separation, consequently, the “viscosity” of crowded suspensions increases.

It is concluded that a correct combination of the viscosity of matrix phase and the granulometry of the aggregates phase can lead to a homogeneous flow. Allowing the prediction of rheological behavior of crowded suspension according to the required needs.

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