

Investigation of the effect of consolidation on cement flow behaviour

Turki, D., Saidani, M., Belarbi, E-H. & Fatah, N.

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15	8	
16 17	9	Djamel TURKI, Dr.
18 19	10	Univ-Tiaret, Laboratoire de Génie électrique et des plasmas, Tiaret, Algeria.
20 21	11	Messaoud SAIDANI, Dr.
22	12	Coventry University, Faculty of Engineering and Computing, Coventry, UK.
23 24	13	
25	14	El-habib BELARBI, Pr.
26 27	15	Univ-Tiaret, Laboratoire Synthèse et Catalyse, Tiaret, Algeria.
28	16	
29 30	17	Nouria FATAH, Pr.
31 32	18	Unité de Catalyse et Chimie du Solide, UMR CNRS 8181, ENSC Lille, France.
33	19	
34 35	20	
36	21	
37 38	22	Correspondence address:
39 40	23	Dr. Djamel TURKI
41	24	Université Ibn-Khaldoun deTiaret,
42	25	Laboratoire de Génie électrique et des plasmas,
44 45	26	BP 78, 14000 Tiaret,
46	27	Algeria
47 48	28	Tel.:213664119700
49	29	Fax: 21346410225
50 51	30	e-mail: turkidjamel@yahoo.fr
52 53	31	
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Abstract

One of the main problems affecting the flow of cement bulk powder is the formation of cohesive arching at the outlet of the hopper, causing blocking of the silo opening and bridge formation. A simple concept is established which outlines these complications. In this context, the interactions of particles lead to a high degree of consolidation of the cement powder and an increase of adhesion force due to the small size and the large surface area of the cement particles. The results from the consolidation test and the flow properties (cohesion) show that the cement powder flow is mainly controlled by internal forces (Van der Waals and adhesion forces) and external forces. These forces have a direct influence on the powder structure, leading to a variable packing behaviour.

Since the problem is due mainly to the interparticle forces, before storage of the cement powder in silo, the powder should be fluidised with air at high velocity to disintegrate the cohesive structure and to overcome over this undesirable property of cement flow.

Keywords: Compressive strength; Modeling; Rheological/rheological properties; Stress; Set-packing.

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8 9	1	Notati	on
10	2	FN	Normal force
11	3	F_{VdW}	Van der Waals force
13	4	Fs	Adhesion force
14	5	А	Hamaker constant
15 16	6	D	Reduced diameter
17	7	z	Distance between the two particles
18 19	, 8	- Sc	Contact surface ($s_c = \pi r^2$)
20	9	r	Radius of the contact surface
21 22	10	, F.	Adhesion force
23	11	P R	Particle radius
24 25	11	ĸ	Poducod Vouna's modulus
26	12	л а	
27 28	13	U	External stress
29	14	<i>a</i> p	
30 31	15	n E	
32	16	F _{ex.}	External force
33 24	17	S	Cell surface
34 35	18	М	Mass of the powder in the cell
36	19	h	Height of the packed bed of particles
37 38	20	(1-ɛ)	Solid fraction
39	21	$ ho_{ m P}$ p	Particle density
40 41	22	T _C	Cohesion
42	23	σ_1	Maximum stress
43 44	24	σ_c	Compressive resistance
45	25	Ni	Number of particles in class <i>i</i>
46 47	26	di	Average diameter of the particles in class i
48	27	FF _c	Flow property.
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1. Introduction

 Several cement plants deal with bulk flow problems that have a detrimental impact on production efficiency, as described by Maynard (2004). Most of the methods used for measuring the flowability of cement make use of some concepts developed in powder mechanics as shown by Maynard (2004). The cement powder flow is mainly controlled by internal and external forces. These forces are the main cause for agglomerating the cement particles in concrete and of resulting of poor flow properties, as discussed by Flatt (2004).

The physical properties of cement powder are directly related to the conception of appropriate and efficient storage equipment as shown by Ganesan et al. (2008). The consolidation and porosity of the solid structure are linked to the understanding of the cement powder flow behaviour, as reported by Leturia et al. (2014). Holdich (2002) stated that the effect that the solid fraction has on flowability powder is probably the most interesting part of the investigation. The powder structure porosity is mainly related to the bulk density which is the combined density of the powder and the void space as shown by Holdich (2002).

The complexity of the cement powder structure requires an examination of the powder behaviour, in particular the particles interaction and pore description. The small size and the large surface area of the cement particles lead to the formation of agglomerates and change the porosity of the solid structure that may be reduced by polymeric dispersants addition, as stated by Uchikawa et al. (1997), Ramachandran et al. (1998) and Aïtcin et al. (1994). The consolidation of the powder would reduce the void of the structure and hence increases the effectiveness and toughness of the material, as described by Li and Kwan (2014). The packed density of the cement powder is a basic aspect governing the

effectiveness of concrete as confirmed by Li and Kwan (2014). The adhesion forces can incite efficiency reduction in the industrial processes, as described by Siegel et al. (1963). Flatt (2004) showed that the flowability of a powder structure is related to the adhesive forces between individual particles.

5 Understanding the behaviour of adhesion interactions between particles and surfaces can 6 contribute to the understanding of the cement powder flow. The different forces involved 7 have to be considered under consolidation namely the Van der Waals, the adhesive and 8 external forces, as was showed by Turki and Fatah (2010). In this context, Flatt (2004) 9 showed that it is essential to evaluate the magnitude of the attractive interparticle forces.

The aim of this research is to present a simple model that takes into account the interparticle forces and the variation of porosity of cement powder bed under external stress (consolidation). That is used to explain the cohesion and the other explicit macroscopic properties of the cement powder flow. The adhesive forces of the cement powder structure are examined and related to the flow obstruction in a silo.

2. Models related to the interparticle forces

Forsyth et al. (2002) elucidated that the interparticle forces between particles of group C Geldart (1973) classification are significant compared to the inertial and gravitational forces, causing poor particle flowability. The adhesion forces would increase with compaction. Schulze (2008) and Tomas (2007) reported that the adhesive forces acting between the particles increase when the particles are constrained to each other by external forces, showing that significant interactions between particles occur, leading to plastic deformation of the particles in the contact region. In this situation Schulze (2008) and Tomas (2007) described that the powder cement flow is principally related to the forces or stresses formerly acting on the powder structure. These forces comprise the consolidation stress exerted on the powder structure, with resulting increase of the adhesion force and hence forming a more compact powder. These concepts rely on interparticle forces estimations that are subject to the appropriate models and assumptions made by Schulze (2008), Tomas (2007).

7 The behaviour of cohesive powders is outlined principally by the contact of external forces 8 acting on the surface of particles and the cohesion due to the interparticle forces (Van der 9 Waals and adhesion forces). Molerus (1975) assumed that during consolidation, the total 10 normal force due to external force is in equilibrium with other forces.

$$11 \qquad F_N = F_{VdW} + F_s$$

12 1.

The Van der Waals force between particles is the main parameter that dominates the powder cohesion as stated by Rumpf (1962), and controls the adhesion between fine particles and, in turn, affects the bulk behaviour of powder. Li et al. (2006) and Tomas (2007) stated that the influence of particle adhesion is defined by surface forces i.e. Van der Waals forces. However, under external stress, particles may deform when in contact with each other as reported by Castellanos (2005).

19 The London-Van der Waals attractive force at solid interface occurring as a result of 20 changing dipoles at the atomic level were integrated by Hamaker (1937) to estimate the 21 attraction between molecules. The Hamaker theory (1937) is used to estimate the Van der 22 Waals force. This force is considered only when the particle surfaces are closer as

confirmed by London (1937). An improved model suggested by Langbein (1969) and Götzinger and Peukert (2003) mainly considers the surface properties of particles.

The Van der Waals force is deduced from the energy interaction between two particles given by Xie (1997) as:

 $F_{VdW} = \frac{AD}{12z^2} \left[1 + \frac{s_c}{\pi Dz} \right]$ 2.

Johnson et al. (1971) showed that *r* is given as:

$$10 \qquad r^3 = \frac{3F_s R}{K}$$

3.

12 The normal force as outlined by Equation (1) can be written as:13

$$F_{N} = \frac{AR}{12z^{2}} \left[1 + \frac{s_{c}}{\pi Rz} \right] + F_{s} = \frac{AR}{12z^{2}} \left[1 + \frac{3^{2/3} F_{s}^{2/3}}{R^{1/3} z K^{2/3}} \right] + F_{s}$$

15 4.

16 The adhesive force F_s in the contact of packed particles resulting from the application of 17 external stress σ is given by Rumph (1962) as:

18
$$F_s = \frac{\sigma \pi d_p^2}{(1-\varepsilon)n}$$

19 5.

20 According to Nakagaki and Sunada (1968), the coordination number *n* is given by:

21
$$n = 1.61\varepsilon^{-1.48} \ (\varepsilon \le 0.82)$$

6.

The adhesive force is expressed with the particle diameter, therefore for different particle size distributions, theoretically, the adhesive force would remain the same if the particleparticle interactions have the same magnitude of interaction in the sample of analysis, but in fact the particle size distribution depends on the arrangement of the particles (particles interactions especially for fine powders categorized in the class C of Geldart (1973) classification. It is complex to calculate the adhesion force for each particle in the particle size distribution. To make the problem less complex, we consider the average diameter (applicable only for a uniform distribution) and assuming that the contact is between two particles that have the same average diameter.

3. Experimental procedure

3.1 Material and methods

The powder used in this work is the ordinary Portland cement (OPC) of class CEM II/A-L 42.5 N.

The relative content of oxide in the cement powder is determined with the use of an energy dispersive micro-XRay Fluorescence spectrometer M4 TORNADO (Bruker). This instrument is equipped with 2 anodes a Rhodium X-ray tube 50 kV/600 mA (30 W) and a Tungsten X-Ray tube 50 kV/700 mA (35 W). For sample characterization, the X-rays Rhodium with a polycapillary lens enabling excitation of an area of 200 µm was used. The measurement was done under vacuum (20 mbar). The elements, that can be measured by this instrument unit range from sodium Na to uranium U. Quantitative analysis was done using fundamental parameter (FP) (standardless). As elements are present in stoichiometric compounds, its formula was used for quantification of the weight percent of each element.

For each sample 36 points (of 200 µm) were analysed, the results are showed as
 elemental and stoichiometric analysis (based on Formula of the oxide). For each sample,
 the mean value and standard deviation are presented in Table 1a.

The physical characteristics of the cement powder according to the Algerian Norms (NA442), which is equivalent to the European Standard EN 197-1:2011, and that used in this study are indicated in Table 1b.

3.1.1 X-ray diffraction characterization

9 X-ray diffraction measurements of the studied cement were performed on a Rigaku 10 Miniflex-600 using SC-70 detector. The powder diffraction patterns of the cement were 11 recorded using Bragg–Brentano geometry and Cu-K α radiation (λ = 1.5406 Å) in the range 12 of 3°–80° 20. A scan rate of 5°/min was used. The Rigaku PDXL 2 software was used to 13 analyze the diffraction pattern.

3. 1.2 Scanning Electron Microscope

16 The microscopic morphology of the Alite particles was examined by using a SEM Hitachi17 SN-3400.

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3.2 Consolidation Test

To estimate the extent of cohesiveness of the cement powder, a consolidation test was carried out to examine the variation of the powder volume and the reduction of the porosity tendency under normal stress.

The consolidation test is reported in a previous article by Turki et al (2015) and was carried out on the cement powder to analyse the variation of the powder volume and the reduction of the porosity under normal stress.

This test is not a measurement of flowability but is associated with various environmental processes, such as storage in hoppers as reported by Leturia et al. (2014). Figure 1 shows the consolidation test as described by Turki et al. (2015). The external stress is written as:

- $\sigma = \frac{F_{ex.}}{S}$
- 8 7.

9 Consequently, the difference of the solid fraction $(1-\varepsilon)$ is considered and correlated to the 10 external stress σ by the relationship:

- $1 \varepsilon = \frac{M}{\rho_{\rm p} Sh}$
- **8**.

15 3.3 Shear Cell

The flow of the powder depends on its consolidation. The effect of consolidation stress on the powder is dependent of the packing and rearrangement of the particles. Accordingly, it is fundamental to assess the flowability of the powder according to the consolidation condition as stated by Diederich et al. (2012). The flowability and cohesion of powder were presented in a previous research, Turki et al. (2015). The measurements of the flowability and cohesion of powders were made with the shear cell of Schulze (1995) as illustrated in Figure 2.

4. Results and discussion 4.1 Cement characterization The Figure 3 shows the diffraction patterns obtained by the X-ray diffraction characterization. The content of the cement is estimated by quantitative analysis of the diffraction patterns Figure 3, based on the reference intensity ratio (RIR) method integrated in the PDXL 2. Software. The different phases present in our sample are presented in Table 2. The X-ray diffraction measurements investigation indicates that the principal silicate phases existing in all the samples are Alite, tricalcium silicate Ca₃O₅Si. To observe the microscopic morphology of the Alite particles, the cement powder was analyzed by scanning electron microscope using a SEM Hitachi SN-3400. Figure 4 shows the polymorphism of the Alite particles and the surfaces geometry. The polymorphism of the Alite particles as shown by Courtial et al. (2003) might have a great impact on the cement powder cohesion. Subsequently, the particle surfaces are closer which enhance the Van der Waals forces interaction Hamaker (1937). As illustrated by SEM photos in Figure 4, surface geometry has an important effect on the interaction between particles. This gives an insight towards modelling these interactions using surface-geometry based models. 4.2 Powder size distribution The powder size distribution illustrated in Figure 5 was measured with a laser light-scattering instrument (Beckman-Coulter, LS230). The average diameter of the cement powder was calculated in accordance to the Sauter diameter definition "surface volume". Explicitly, the average diameter is calculated according to the diameter definition "Surface-Volume" or Sauter diameter as

$$d_p = \frac{\sum N_i d_i^3}{\sum N_i d_i^2}$$

9.

Where *Ni* is the number of particles in class *i* and d_i is the average diameter of the particle in this class.

5 The average diameter was given by the laser light-scattering instrument (Beckman-6 Coulter, LS230), using the Algorithm to compute the Sauter diameter from a large set of 7 data, giving an average diameter of 4.6 µm.

The particle density ρ_p was measured with a helium pycnometer (Micromeritics, AccuPyc 1330) giving a density of 3577 kg/m³. Taking into account the particle density and the average size of the cement powder, the powder can be categorized under group C of the Geldart (1973) classification.

4.3 Powder flow and consolidation

The yield locus and the variation in solid fraction according to the normal stress of the cement powder are illustrated in Figure 6 and Figure 7, respectively. As enlightened by Turki et al. (2015).

The results of flowability and cohesion of powder were presented in a previous research, Turki et al. (2015). Figure 6 indicates that the cohesion τ_c =578 Pa. From the yield locus, the maximum stress σ_1 =5370 Pa and the compressive resistance σ_c =2169 Pa. The parameter FF_c is defined as the ratio between σ_1 and σ_c that defines the flow property. FF_c was found to be 2.48, resulting that the cement powder is classified as cohesive, difficult flow.

During consolidation, the cement powder structure is uniform and formed by a number of

- **23**

particles in contact. The variation of the solid fraction is mainly due to the interparticle
 forces and tends to attain a linear regime, Figure 7.

From the consolidation test and using the models developed in Equation (4) and Equation (5). With a Hamaker constant *A* of Ordinary Portland Cement (OPC) as 1.72 × 10⁻²⁰ J. given by Lomboy et al. (2011) and the cement particle diameter of 4.6µm. The distance between particle surfaces *z* is taken as 4×10⁻¹⁰ m, according to Krupp (1967). The reduced Young's modulus is given by Boumiz et al. (1997) as 117.6 GPa.

The variation of the adhesion force F_s to the normal force F_N obtained from Equation (4) is illustrated in Figure 8. The adhesive force starts increasing linearly with the normal force, showing that at initial stage important air diffusion occurs within the powder structure, with the contact number between particles arising from the adhesion forces. Then the particles start to be set in a compact arrangement. Subsequently, a strong cohesion between particles occurs, leading to the formation of an important number of agglomerates. This confirms that the behaviour of cement powder under consolidation is controlled by the internal forces.

For higher values of adhesion force, the transition region is attained resulting in an increase of solid fraction and attainment of a linear regime. The load is extended on the solid structure. Thus, there is consolidation of the packed bed of powder. Consequently, the cement powder flow is mainly affected by the adhesion forces and internal forces due to the time consolidation of cement in the silo as stated by Schulze (1995). As the adhesion force increases, the impact of the Van der Waals forces on the behaviour becomes considerable in forming a large number of agglomerates as illustrated in Figure 9 using Equation (2). Confirming that the interaction between particles is important and this validates the results of the shear test as stated by Turki et al. (2015). Consequently, taking

into account the flow of the cement powder in a silo without consolidation, the powder is subjected to its weight that plays the role of the adhesive forces, involving an increase in the Van der Waals forces, hence causing a larger cohesion between particles and then generating a clogging at the silo outlet.

5 This behaviour is confirmed in Figure 10, showing the evolution of the solid fraction 6 according to the adhesion force where the first part of the graph increases linearly with a 7 straight up development, showing an enhancement in the solid fraction by evacuating the 8 air in the powder structure.

Figure 10 is deduced from the consolidation test resulting in determining the solid fraction (1- ε) and a combination of the adhesion force F_s given by the equation of Rumph (1962), equation 5. Then, the structure is formed of a partial uniform powder. This behaviour is expected to reflect a disintegration of the agglomerates and an increase of the contact surfaces between the particles. The solid fraction-adhesion force curve shows a large upward change of the solid fraction to give a more stable solid structure.

This research revealed that the consolidation test and the flow properties highlight that the cohesion of cement powder is controlled by internal forces and external forces. Throughout the exertion of external forces, different interactions take place in the powder structure. Furthermore, it is interesting to find the relationship between the impact of the Van der Waals forces acting as an isolated interaction to the adhesive forces, that highlights the powders' particle to agglomerate under the action of the powder weight and, therefore, stopping the free flowing of the cement powder.

Similarly, Figure 8 and Figure 10 show the variation of the adhesive and Van der Waals
 forces with the normal force and the solid fraction respectively, resulting from the
 consolidation test and using the force equilibrium model of Molerus (1975). These figures

highlight the considerable role of the internal forces in forming cohesive arching at the
 outlet of the hopper.

The deformation of the contact surfaces between the particles facilitates the increase of the Van der Waals forces, resulting in an expansion of the contact region, as confirmed by Krupp (1967). This deformation is due to the adhesion forces; ensuing an enhancement of particles cohesion primarily due to consolidation.

5. Conclusion

9 Understanding the relationship between the cement powder flow and the adhesion forces 10 is the way to overcome the obstruction for the cement powder flow. Cement powder flow 11 was investigated and quantified by using various techniques such as shear stress and 12 consolidation.

13 The interparticle forces are at the origin of the formation of arches at the base of the silo 14 mainly due to an increase in adhesion forces. The cement powder has a tendency to 15 consolidate to form a more compact structure and therefore hinder the flow.

16 The results from the consolidation test and the flow properties (cohesion) show that the

17 cement powder flow is mainly controlled by internal forces (Van der Waals and

18 adhesion forces) and external forces.

19 As these results confirm the blocking of the silo opening a further research would be

20 carried out, aiming to disintegrate the cohesive structure by fluidisation of the cement

 $\frac{2}{2}$ 21 powder with air before undertaking the powder stockage in silo. The fluidisation process of

cohesive cement powder would lead to the suspension of the agglomerates (made up of primary particles) at a very high gas velocity (above the minimum velocity). The cement powder is mainly composed of polymorphism Alite particles which could be assessed by a number of asperities in contact. These in turn will enhance the Van der Waals forces interaction, which is an interesting area of research that needs to be further, investigated. References Aïtcin PC, Jolicoeur C, MacGregor JG (1994) The reology of cementitious materials. Concrete International. 16(5): 45. Boumiz A, Sorrentino D, Vernet C et al. (1997) Modelling the development of the elastic moduli as a function of the degree of hydration of cement pastes and mortars, in Proceedings 13 of the 2nd RILEM Workshop on Hydration and Setting: Why does cement set? An interdisciplinary approach, edited by A. Nonat (RILEM Dijon, France). Castellanos A (2005) The relationship between attractive interparticle forces and bulk behaviour in dry and uncharged fine powders. Advances in Physics. 54 (4): .263-376. Courtial M, de Noirfontaine MN, Dunstetter F et al. (2003) Polymorphism of tricalcium silicate in Portland cement: a fast visual identification of structure and superstructure. Powder Diffr. 18(1): 7-15. Diederich P, Moureta M, Ryckc A et al. (2012) The nature of limestone filler and self-consolidating feasibility-Relationships between physical, chemical and mineralogical properties of fillers and the flow at different states, from powder to cement-based suspension. Powder Technology. 218: 90-101. EN 197-1 (2011) Cement-Part 1: Composition, specifications and conformity criteria for common cements. Flatt RJ (2004) Dispersion forces in cement suspensions. Cement and Concrete Research. 34(3): 399-408. Forsyth AJ, Hutton S, Rhodes MJ (2002) Effect of cohesive interparticle force on the flow

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16 17		
18 19 20		
21 22 23		
24 25		
26 27 28		
29 30 31		
32 33		
34 35 36		
37 38 39		
40 41		
42 43 44		
45 46 47		
48 49 50		
50 51 52		
53 54 55		
56 57		
59 60		
61 62 63		
64 65		



figieH bea



Figure 2



Intensity (CPS)









(ed) 1









Chemical composition of cement CEM II/A-L 42.5 N			
Component	Mean value (%)	Standard deviation	
Fe ₂ O ₃	2.822578	0.068299	
K ₂ O	0.782745	0.029200	
MgO	0.814106	0.283107	
S _i O ₂	14.56387	0.432004	
Al ₂ O ₃	3.264997	0.148407	
Na ₂ O	0.000394	0.002587	
CaO	73.47939	0.473208	
SO ₃	3.902756	0.347077	
T _i O ₂	0.242009	0.013455	

Physical and mechanical properties of cement CEM II/A-L 42.5 N				
Initial setting	Initial setting time (min)		125	
Final setting t	time (min)	185		
Thermal expan	nsion (mm)	0.47		
Specific area (Bl	aine) (cm ² /g)	4465		
Standard consist	ency (%H ₂ O)	27.04		
	Compressive stre	ngth (MPa)	Flexural strength (MPa)	
2 days	23.19		4.99	
7 days	35.24		6.78	
28 days	44.64		7.80	

Phase name	Content (%)	
Alite	51	
Brownmillerite	15	
Periclase	11.7	
Ferrite	9.2	
Quartz	5.4	
Belite (Dicalcium Silicate)	4.7	
Portlandite	1	
Arcanite	1	
Lime	0.8	

Table 1a. Micro-XRay Fluorescence spectrometer analysis of the cement class CEM II/A-L 42.5 N

- Table 1b. Physical characteristics of cement class CEM II/A-L 42.5 N conforming to NA442 (or EN 197-1:2011) See:(https://www.scimat.dz/portail/gamme/ciments/)
- Figure 1. Consolidation test
- Figure 2. Annular shear cell Schulze (1995)
- Figure 3. Diffraction patterns of Portland cement (OPC) of class CEM II/A-L 42.5 N
- Table 2. Quantitative analysis of Portland cement (OPC) of class CEM II/A-L 42.5 N
- Figure 4. SEM photos of cement (OPC) of class CEM II/A-L 42.5 N, scaling (100 µm, 20 µm, 10 µm and 5µm)
- Figure 5. Particle size distribution of cement powder
- Figure 6. The Mohr's circle and yield locus of cement powder Turki et al. (2015)
- Figure 7. Variation in solid fraction according to the normal stress for cement powder Turki et al. (2015)
- Figure 8. Variation of the adhesion and Van der Waals forces vs. the normal force

Figure 9. The variation of Van der Waals forces to the adhesion force

Figure 10. The evolution of the solid fraction according to the adhesion force