

# Investigation of the effect of consolidation on cement flow behaviour

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

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8  
9 Djamel TURKI, Dr.  
10 Univ-Tiaret, Laboratoire de Génie électrique et des plasmas, Tiaret, Algeria.  
11  
12 Messaoud SAIDANI, Dr.  
13  
14 Coventry University, Faculty of Engineering and Computing, Coventry, UK.  
15  
16  
17 El-habib BELARBI, Pr.  
18  
19 Univ-Tiaret, Laboratoire Synthèse et Catalyse, Tiaret, Algeria.  
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22 Nouria FATAH, Pr.  
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**Correspondence address:**  
Dr. Djamel TURKI  
Université Ibn-Khaldoun deTiaret,  
Laboratoire de Génie électrique et des plasmas,  
BP 78, 14000 Tiaret,  
Algeria  
Tel.:213664119700  
Fax: 21346410225  
e-mail: turkidjamel@yahoo.fr

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1     **Abstract**

2     One of the main problems affecting the flow of cement bulk powder is the formation of  
3     cohesive arching at the outlet of the hopper, causing blocking of the silo opening and  
4     bridge formation. A simple concept is established which outlines these complications. In  
5     this context, the interactions of particles lead to a high degree of consolidation of the  
6     cement powder and an increase of adhesion force due to the small size and the large  
7     surface area of the cement particles. The results from the consolidation test and the flow  
8     properties (cohesion) show that the cement powder flow is mainly controlled by internal  
9     forces (Van der Waals and adhesion forces) and external forces. These forces have a  
10    direct influence on the powder structure, leading to a variable packing behaviour.  
11    Since the problem is due mainly to the interparticle forces, before storage of the cement  
12    powder in silo, the powder should be fluidised with air at high velocity to disintegrate the  
13    cohesive structure and to overcome over this undesirable property of cement flow.

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

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1	<b>Notation</b>	
2	$F_N$	Normal force
3	$F_{VdW}$	Van der Waals force
4	$F_s$	Adhesion force
5	$A$	Hamaker constant
6	$D$	Reduced diameter
7	$z$	Distance between the two particles
8	$s_c$	Contact surface ( $s_c = \pi r^2$ )
9	$r$	Radius of the contact surface
10	$F_s$	Adhesion force
11	$R$	Particle radius
12	$K$	Reduced Young's modulus
13	$\sigma$	External stress
14	$d_p$	Average particle diameter
15	$n$	Coordination number
16	$F_{ex.}$	External force
17	$S$	Cell surface
18	$M$	Mass of the powder in the cell
19	$h$	Height of the packed bed of particles
20	$(1-\varepsilon)$	Solid fraction
21	$\rho_p$	Particle density
22	$\tau_c$	Cohesion
23	$\sigma_1$	Maximum stress
24	$\sigma_c$	Compressive resistance
25	$N_i$	Number of particles in class $i$
26	$d_i$	Average diameter of the particles in class $i$
27	$FF_c$	Flow property.
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1     **1. Introduction**

2     Several cement plants deal with bulk flow problems that have a detrimental impact on  
3     production efficiency, as described by Maynard (2004). Most of the methods used for  
4     measuring the flowability of cement make use of some concepts developed in powder  
5     mechanics as shown by Maynard (2004). The cement powder flow is mainly controlled by  
6     internal and external forces. These forces are the main cause for agglomerating the  
7     cement particles in concrete and of resulting of poor flow properties, as discussed by Flatt  
8     (2004).

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

17    The complexity of the cement powder structure requires an examination of the powder  
18    behaviour, in particular the particles interaction and pore description. The small size and  
19    the large surface area of the cement particles lead to the formation of agglomerates and  
20    change the porosity of the solid structure that may be reduced by polymeric dispersants  
21    addition, as stated by Uchikawa et al. (1997), Ramachandran et al. (1998) and Aitcin et al.  
22    (1994). The consolidation of the powder would reduce the void of the structure and hence  
23    increases the effectiveness and toughness of the material, as described by Li and Kwan  
24    (2014). The packed density of the cement powder is a basic aspect governing the

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1 effectiveness of concrete as confirmed by Li and Kwan (2014). The adhesion forces can  
2 incite efficiency reduction in the industrial processes, as described by Siegel et al. (1963).  
3 Flatt (2004) showed that the flowability of a powder structure is related to the adhesive  
4 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.  
10 The aim of this research is to present a simple model that takes into account the  
11 interparticle forces and the variation of porosity of cement powder bed under external  
12 stress (consolidation). That is used to explain the cohesion and the other explicit  
13 macroscopic properties of the cement powder flow. The adhesive forces of the cement  
14 powder structure are examined and related to the flow obstruction in a silo.

15

16 **2. Models related to the interparticle forces**

17 Forsyth et al. (2002) elucidated that the interparticle forces between particles of group C  
18 Geldart (1973) classification are significant compared to the inertial and gravitational  
19 forces, causing poor particle flowability. The adhesion forces would increase with  
20 compaction. Schulze (2008) and Tomas (2007) reported that the adhesive forces acting  
21 between the particles increase when the particles are constrained to each other by  
22 external forces, showing that significant interactions between particles occur, leading to  
23 plastic deformation of the particles in the contact region. In this situation Schulze (2008)

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1 and Tomas (2007) described that the powder cement flow is principally related to the  
2 forces or stresses formerly acting on the powder structure. These forces comprise the  
3 consolidation stress exerted on the powder structure, with resulting increase of the  
4 adhesion force and hence forming a more compact powder. These concepts rely on  
5 interparticle forces estimations that are subject to the appropriate models and assumptions  
6 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.

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

12 1.

13 The Van der Waals force between particles is the main parameter that dominates the  
14 powder cohesion as stated by Rumpf (1962), and controls the adhesion between fine  
15 particles and, in turn, affects the bulk behaviour of powder. Li et al. (2006) and Tomas  
16 (2007) stated that the influence of particle adhesion is defined by surface forces i.e. Van  
17 der Waals forces. However, under external stress, particles may deform when in contact  
18 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

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

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

5  
6 
$$F_{vdw} = \frac{AD}{12z^2} \left[ 1 + \frac{s_c}{\pi Dz} \right]$$

7 2.  
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9 Johnson et al. (1971) showed that  $r$  is given as:

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

11 3.

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

13  
14 
$$F_N = \frac{AR}{12z^2} \left[ 1 + \frac{s_c}{\pi Rz} \right] + F_s = \frac{AR}{12z^2} \left[ 1 + \frac{3^{2/3} F_s^{2/3}}{R^{1/3} z K^{2/3}} \right] + F_s$$

15 4.

16 The adhesive force  $F_s$  in the contact of packed particles resulting from the application of  
17 external stress  $\sigma$  is given by Rumpf (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 
$$n = 1.61\varepsilon^{-1.48} \quad (\varepsilon \leq 0.82)$$

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

### 11 **3. Experimental procedure**

#### 12 **3.1 Material and methods**

13 The powder used in this work is the ordinary Portland cement (OPC) of class CEM II/A-L  
14 42.5 N.

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

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1 For each sample 36 points (of 200  $\mu\text{m}$ ) were analysed, the results are showed as  
2 elemental and stoichiometric analysis (based on Formula of the oxide). For each sample,  
3 the mean value and standard deviation are presented in Table 1a.

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

7

### 8 **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 $\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ) in the range  
12 of  $3^\circ\text{--}80^\circ$   $2\theta$ . A scan rate of  $5^\circ/\text{min}$  was used. The Rigaku PDXL 2 software was used to  
13 analyze the diffraction pattern.

14

### 15 **3. 1.2 Scanning Electron Microscope**

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

18

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### 20 **3.2 Consolidation Test**

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22 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.

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

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

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

8 7.

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

11 
$$1-\varepsilon = \frac{M}{\rho_p Sh}$$

12 8.

15 **3.3 Shear Cell**

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

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1 **4. Results and discussion**

2 **4.1 Cement characterization**

3 The Figure 3 shows the diffraction patterns obtained by the X-ray diffraction  
4 characterization.

5 The content of the cement is estimated by quantitative analysis of the diffraction patterns

6 Figure 3, based on the reference intensity ratio (RIR) method integrated in the PDXL 2.

7 Software. The different phases present in our sample are presented in Table 2.

8 The X-ray diffraction measurements investigation indicates that the principal silicate  
9 phases existing in all the samples are Alite, tricalcium silicate  $\text{Ca}_3\text{O}_5\text{Si}$ .

10 To observe the microscopic morphology of the Alite particles, the cement powder was  
11 analyzed by scanning electron microscope using a SEM Hitachi SN-3400. Figure 4 shows  
12 the polymorphism of the Alite particles and the surfaces geometry.

13 The polymorphism of the Alite particles as shown by Courtial et al. (2003) might have a  
14 great impact on the cement powder cohesion. Subsequently, the particle surfaces are  
15 closer which enhance the Van der Waals forces interaction Hamaker (1937). As illustrated  
16 by SEM photos in Figure 4, surface geometry has an important effect on the interaction  
17 between particles. This gives an insight towards modelling these interactions using  
18 surface-geometry based models.

19 **4.2 Powder size distribution**

20 The powder size distribution illustrated in Figure 5 was measured with a laser light-  
21 scattering instrument (Beckman-Coulter, LS230).

22 The average diameter of the cement powder was calculated in accordance to the Sauter  
23 diameter definition "surface volume". Explicitly, the average diameter is calculated  
24 according to the diameter definition "Surface-Volume" or Sauter diameter as  
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$$d_p = \frac{\sum N_i d_i^3}{\sum N_i d_i^2}$$

2 9.

3 Where  $N_i$  is the number of particles in class  $i$  and  $d_i$  is the average diameter of the particle  
4 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  $\mu\text{m}$ .

8 The particle density  $\rho_p$  was measured with a helium pycnometer (Micromeritics, AccuPyc  
9 1330) giving a density of 3577  $\text{kg/m}^3$ . Taking into account the particle density and the  
10 average size of the cement powder, the powder can be categorized under group C of the  
11 Geldart (1973) classification.

### 13 **4.3 Powder flow and consolidation**

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

17 The results of flowability and cohesion of powder were presented in a previous research,  
18 Turki et al. (2015). Figure 6 indicates that the cohesion  $\tau_c=578$  Pa. From the yield locus,  
19 the maximum stress  $\sigma_1=5370$  Pa and the compressive resistance  $\sigma_c=2169$  Pa. The  
20 parameter  $FF_c$  is defined as the ratio between  $\sigma_1$  and  $\sigma_c$  that defines the flow property.  $FF_c$   
21 was found to be 2.48, resulting that the cement powder is classified as cohesive, difficult  
22 flow.

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

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

3 From the consolidation test and using the models developed in Equation (4) and Equation  
4 (5). With a Hamaker constant  $A$  of Ordinary Portland Cement (OPC) as  $1.72 \times 10^{-20}$  J.  
5 given by Lomboy et al. (2011) and the cement particle diameter of  $4.6\mu\text{m}$ . The distance  
6 between particle surfaces  $z$  is taken as  $4 \times 10^{-10}$  m, according to Krupp (1967). The reduced  
7 Young's modulus is given by Boumiz et al. (1997) as 117.6 GPa.

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

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

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1 into account the flow of the cement powder in a silo without consolidation, the powder is  
2 subjected to its weight that plays the role of the adhesive forces, involving an increase in  
3 the Van der Waals forces, hence causing a larger cohesion between particles and then  
4 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.

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

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

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

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

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

7

## 8 **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  
21 powder with air before undertaking the powder stockage in silo. The fluidisation process of



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1 cohesive cement powder would lead to the suspension of the agglomerates (made up of  
2 primary particles) at a very high gas velocity (above the minimum velocity).  
3 The cement powder is mainly composed of polymorphism Alite particles which could be  
4 assessed by a number of asperities in contact. These in turn will enhance the Van der  
5 Waals forces interaction, which is an interesting area of research that needs to be further,  
6 investigated.

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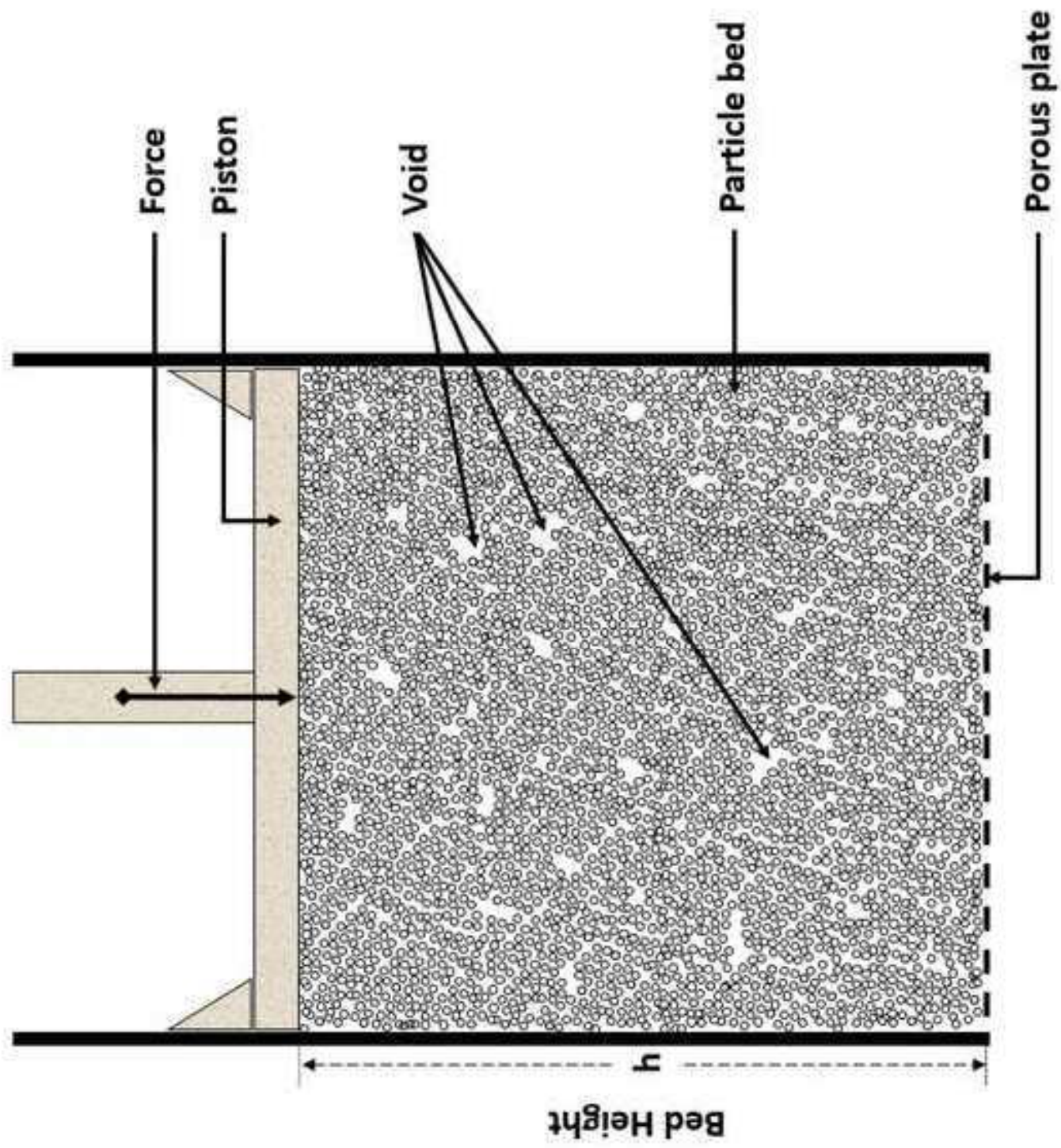


Figure 1

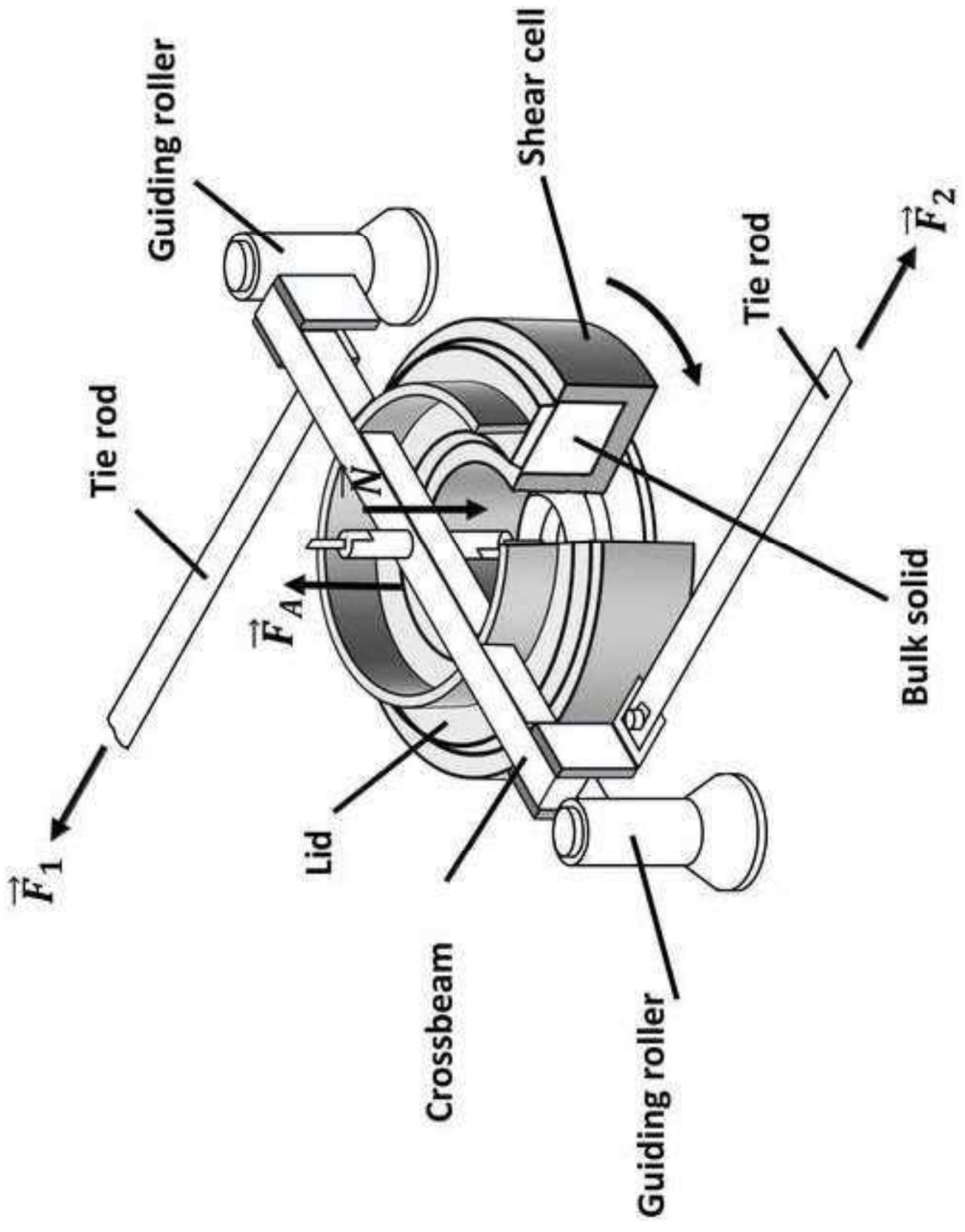


Figure 2

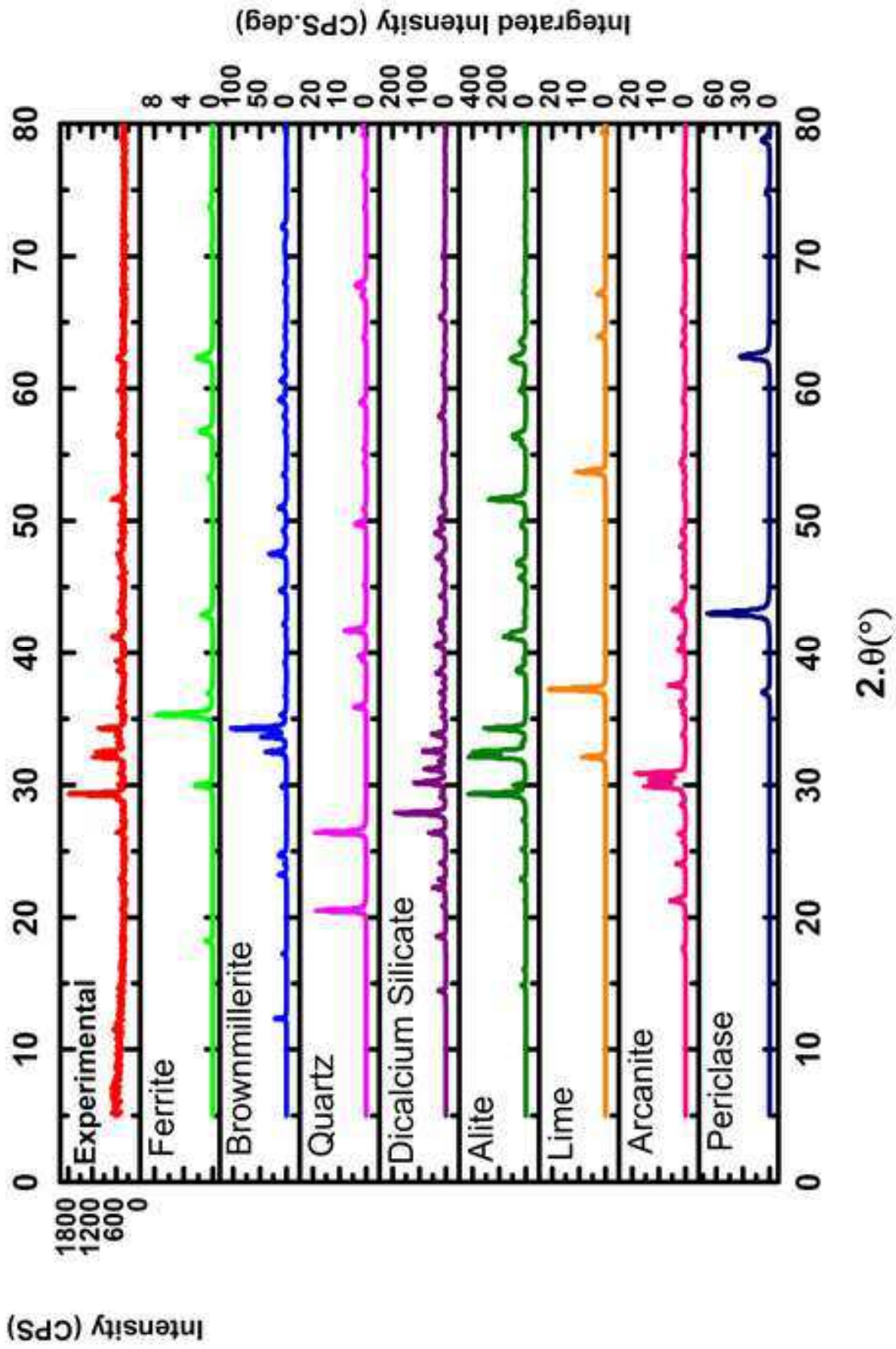
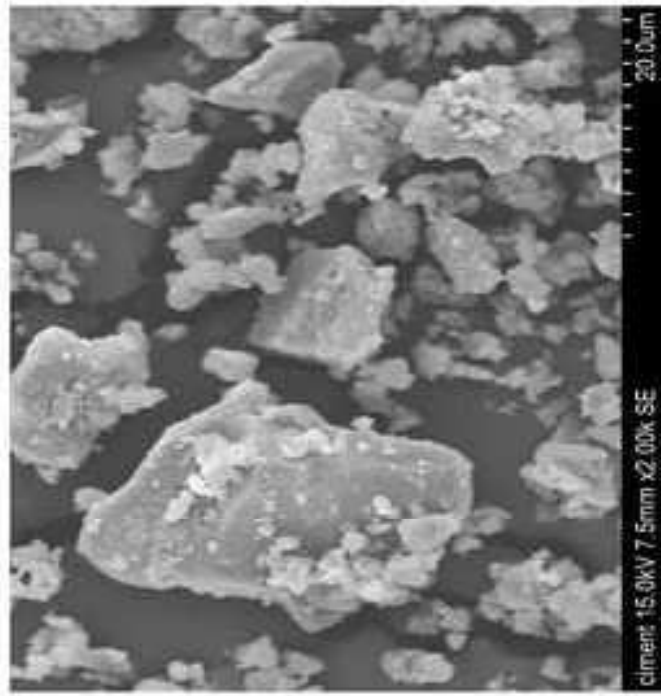
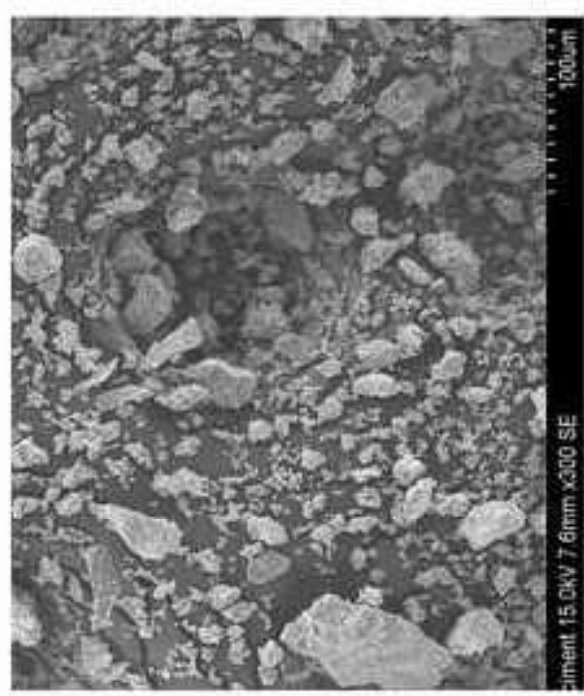
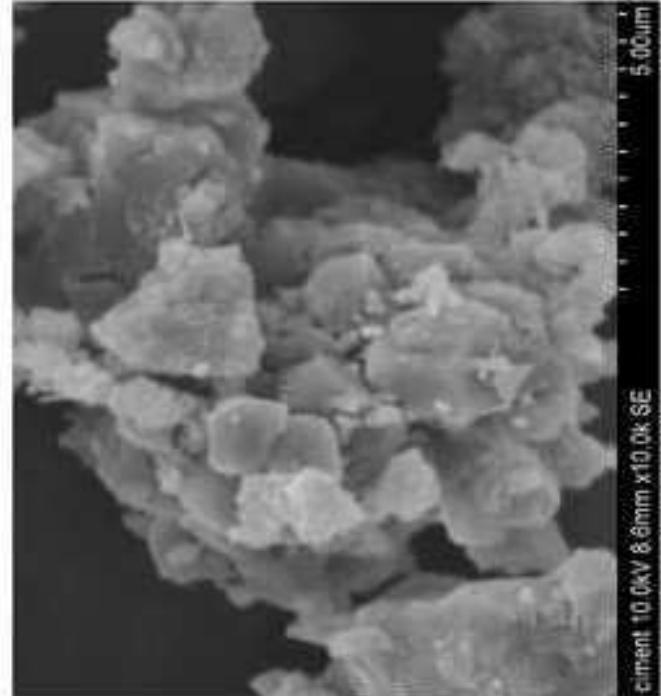
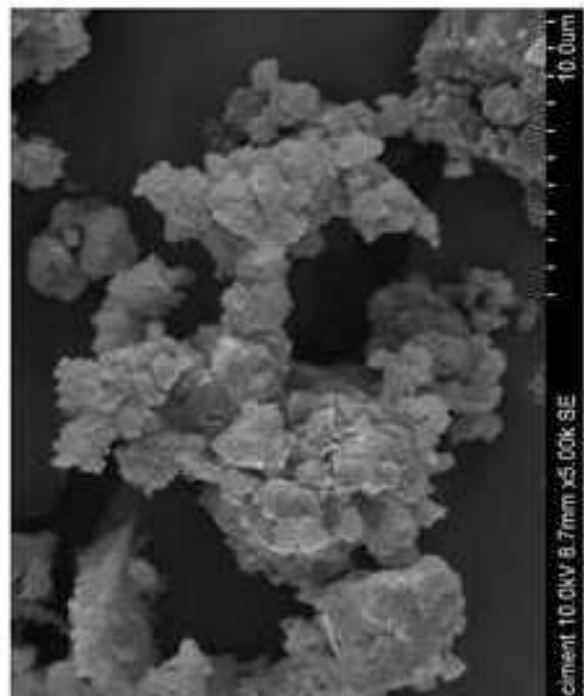


Figure 3

Figure 4





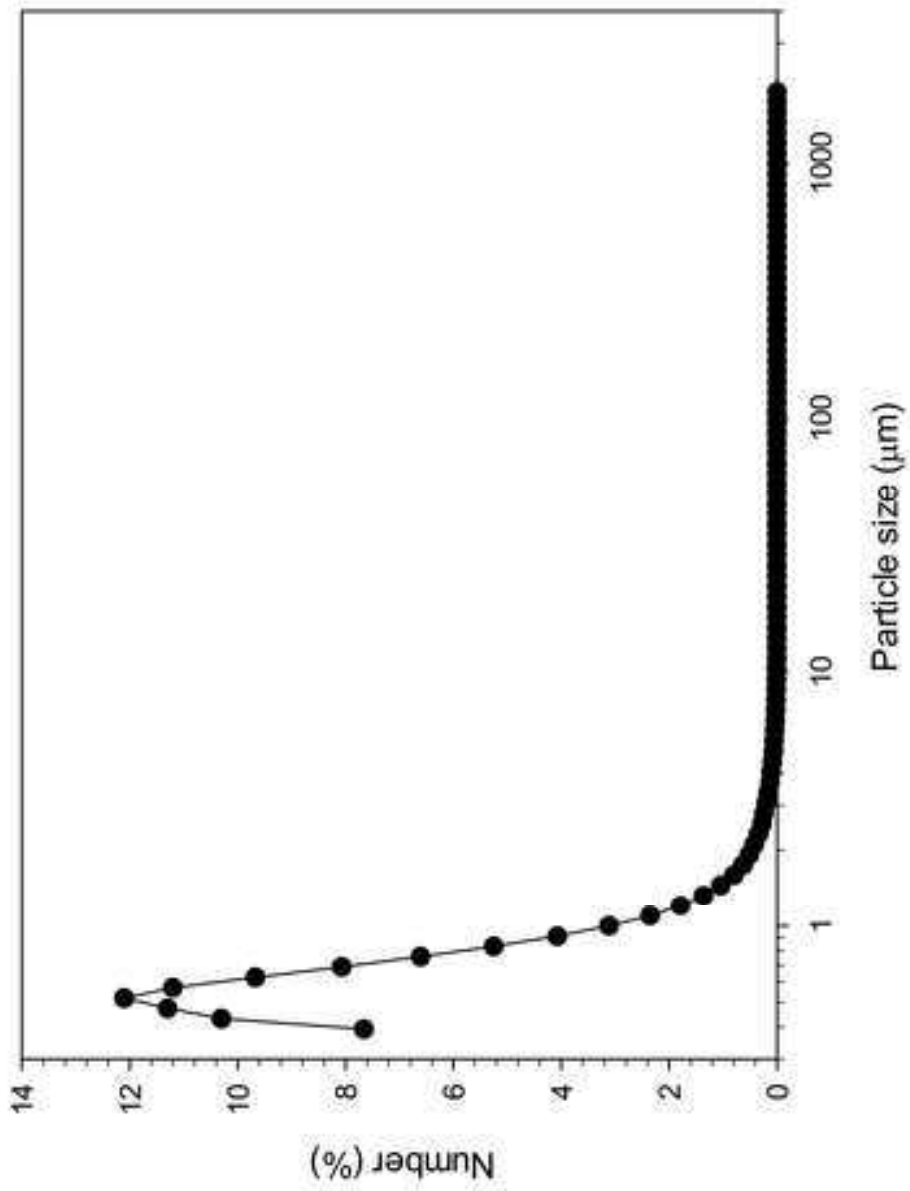


Figure 5

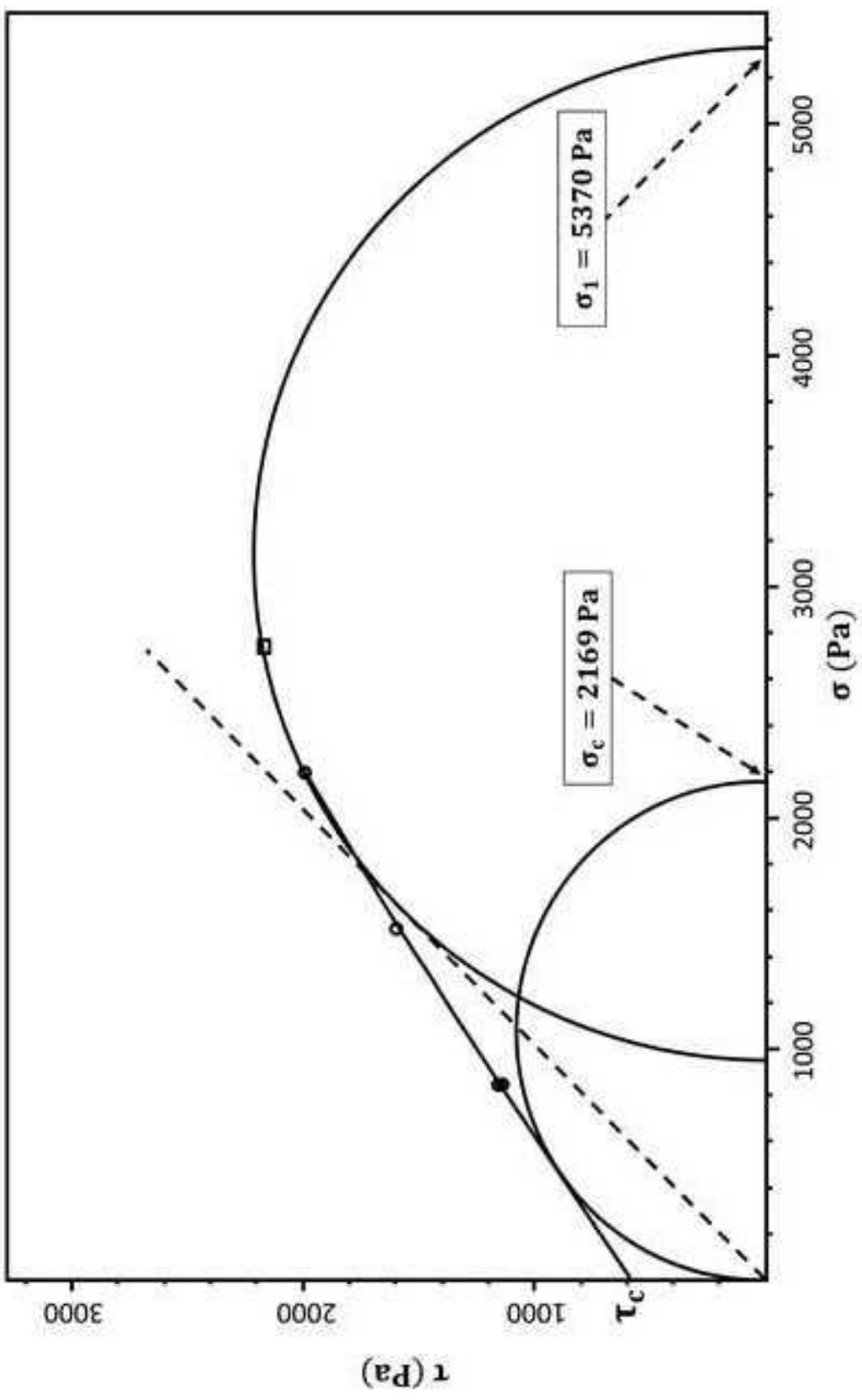


Figure 6

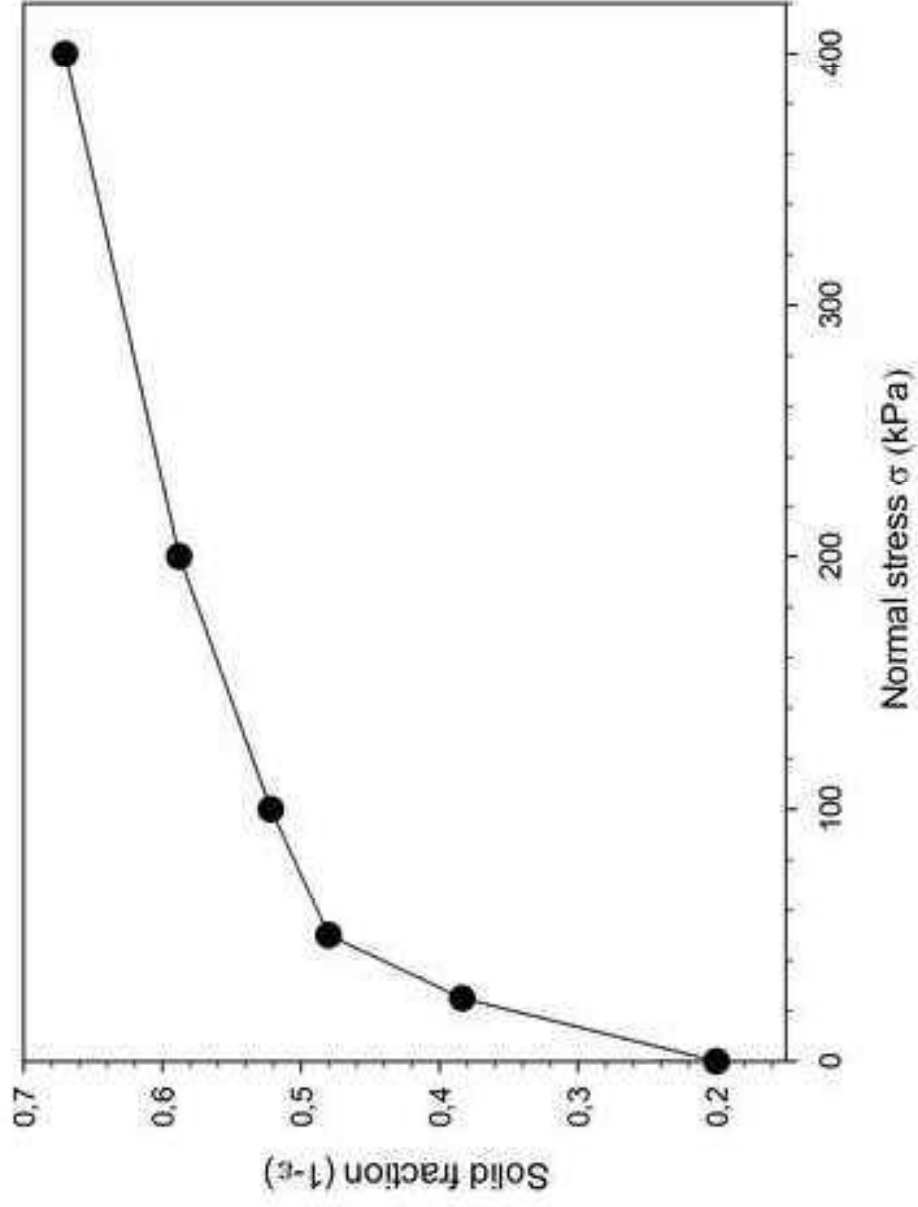


Figure 7

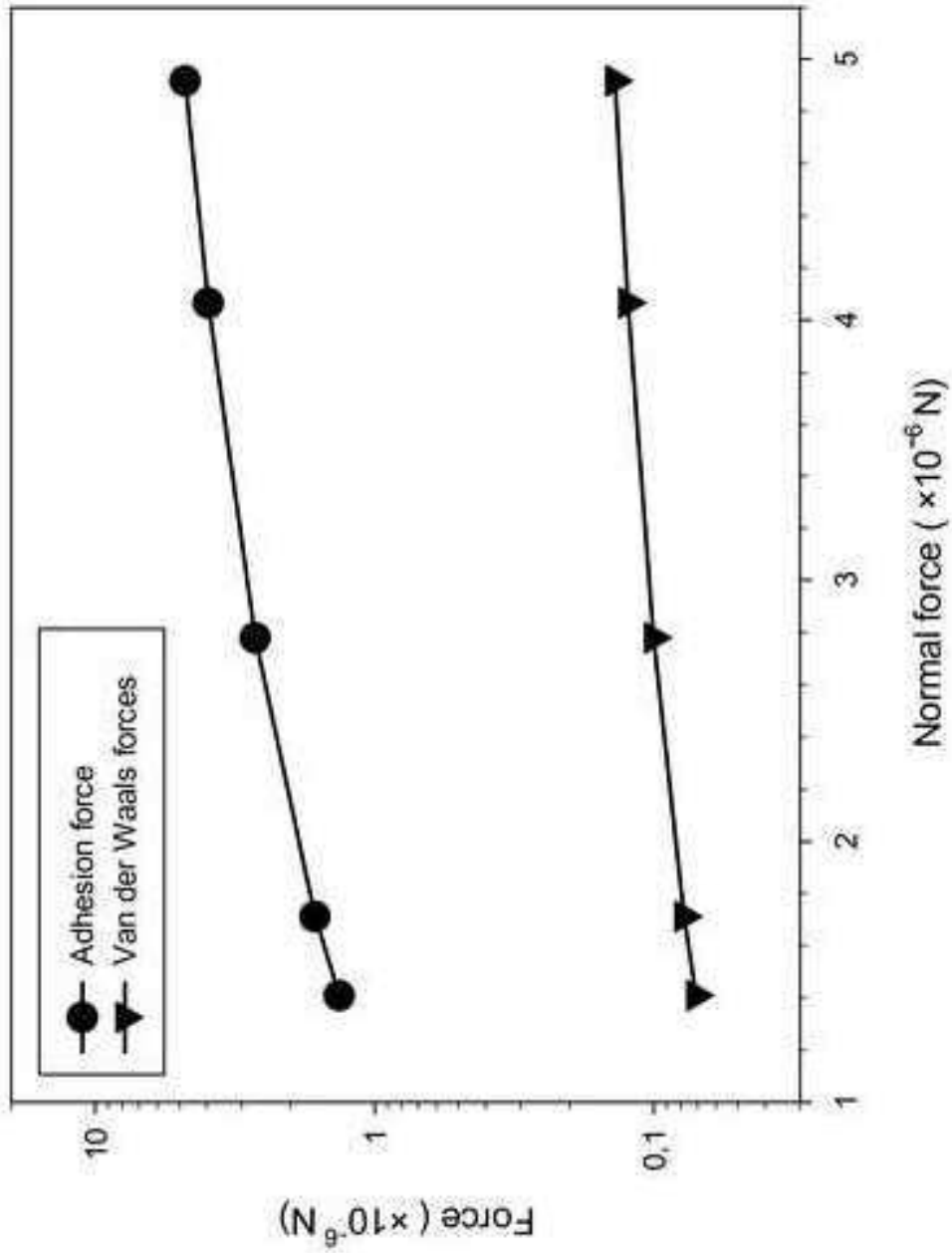


Figure 8

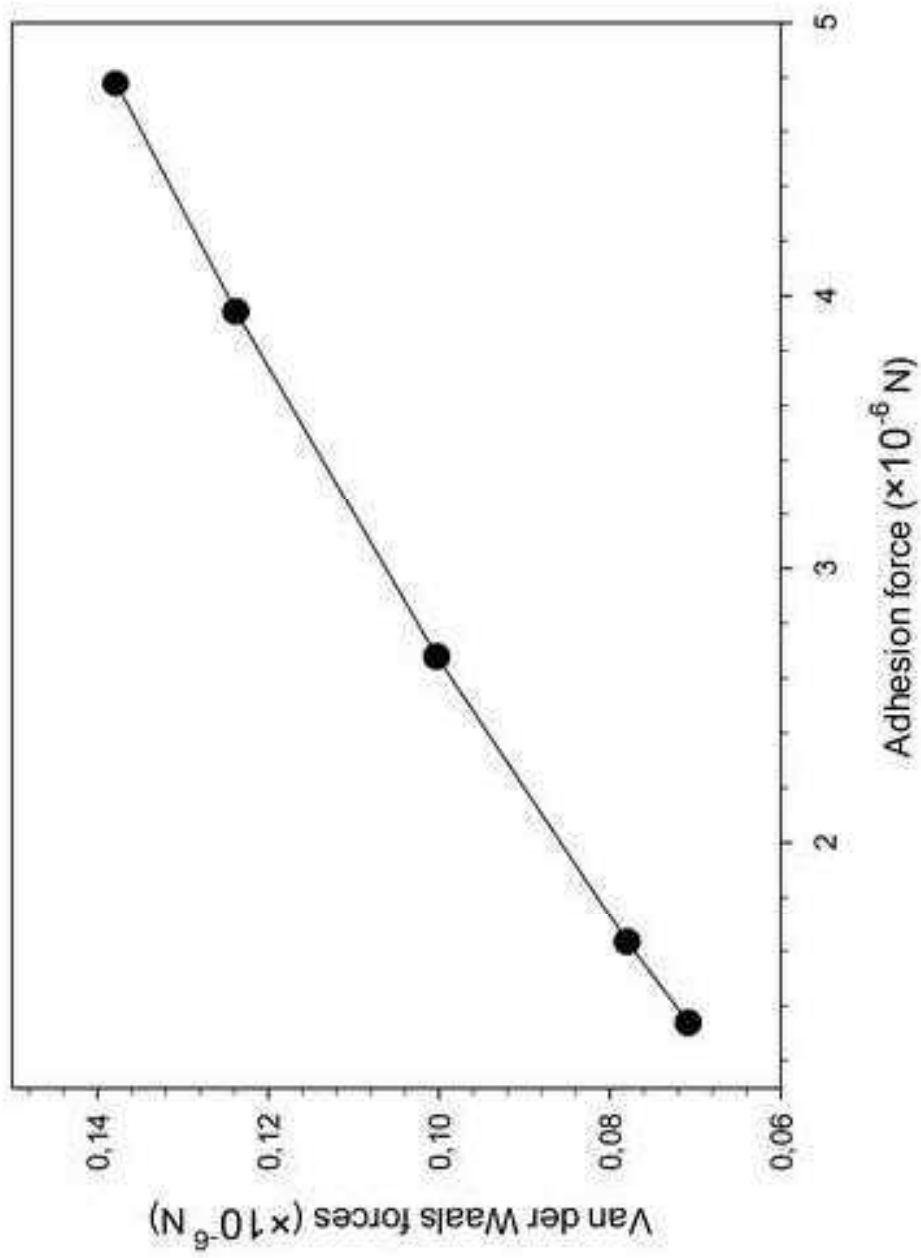


Figure 9

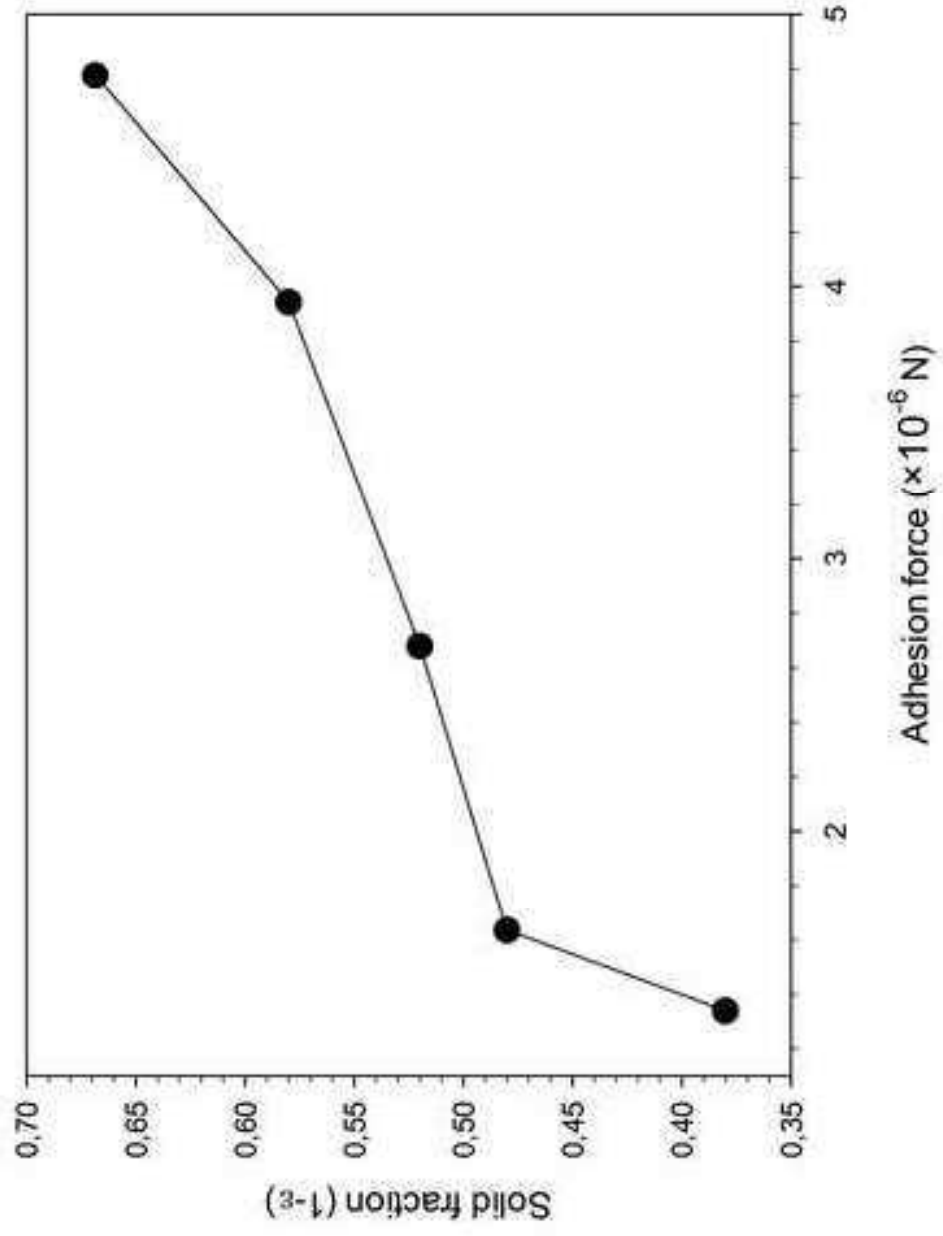


Figure 10

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

<b>Physical and mechanical properties of cement CEM II/A-L 42.5 N</b>		
Initial setting time (min)		125
Final setting time (min)		185
Thermal expansion (mm)		0.47
Specific area (Blaine) (cm <sup>2</sup> /g)		4465
Standard consistency (%H <sub>2</sub> O)		27.04
	<b>Compressive strength (MPa)</b>	<b>Flexural strength (MPa)</b>
<b>2 days</b>	23.19	4.99
<b>7 days</b>	35.24	6.78
<b>28 days</b>	44.64	7.80



Table 2

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  $\mu\text{m}$ , 20  $\mu\text{m}$ , 10  $\mu\text{m}$  and 5 $\mu\text{m}$ )

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