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Rheological Behaviour of Raw Cement

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Abstract: A series of rheometric tests was conducted on raw cement at different concentrations and particle finesses as well. The paste of raw cement, formed by a set of particles suspensions in water, presents rheological properties that make it different from the fluids commonly used. The obtained results have showed that the rheological behaviour is the shear-thinning type and can be described satisfactorily by a model of Herschel-Bulkley, characterized by three parameters τ_0, K and n which connect the shear stress to the shear rate. In addition, the parameter τ_0 was correlated by an exponential function of the volume concentration of particles, this correlation is valid for both the raw cement and limestone. This latter is the main component of raw cement, for that reason its rheology has been studied.

Key words: Limestone, raw meal, rheology, yield stress, solids concentration, particle size

INTRODUCTION

The knowledge of rheology of raw cement is important in the design of milling devices, transport and storage in the industrial manufacturing humid and semi humid cement process. In the wet process, the raw materials, properly proportioned, are then ground with water, thoroughly mixed and fed into the kiln in the form of slurry (containing enough water to make it fluid). Physically, the wet raw cement can be considered as a concentrated suspension of particles of various sizes inside water. To characterize the behaviour of a suspension, some authors have been interested in viscosity. The first classic model for predicting the viscosity of suspensions was based on the law of Einstein (1906):

$$\eta = \eta_0 + (1 + 2,5\,\phi_v) \tag{1}$$

where, η is the dynamic viscosity of the suspension, η_0 is the dynamic viscosity of the liquid and ϕ_v is the volume concentration of particles. This relation is valid only for the weak concentrations (ϕ_v<0.02) and does not take into account the interaction between the particles.

In the range of the more concentrated suspensions, the viscosity of the suspensions is not any more controlled by the viscosity of the fluid and its solids concentration but depends on the maximum compactness of granular stacking ϕ_m. By a theoretical approach, Mooney (1951) proposes for the determination of the viscosity, the following relation:

$$\eta = \eta_0 \exp\left(\frac{2,5\phi_v}{1 - \dfrac{\phi_v}{\phi_m}}\right) \tag{2}$$

Another semi empirical relation, derived from Eq. 2 was proposed by Quémada (1977):

$$\eta = \eta_0 \left(1 - \frac{\phi_v}{\phi_m}\right)^{-2} \tag{3}$$

Several studies were undertaken, aiming to improve predictions of viscosity by refining the methods of calculating of ϕ_m (Berli *et al*., 2005; Guillemin *et al*., 2007). The inter-particle effects have been studied by Usui *et al*. (2001) using the suspensions of silica, It was verified experimentally that the Simha's model was able to predict the slurry viscosity for the case of suspensions with particle size distribution. Toutou *et al*. (2004) have proposed a model of multi-scale viscosity for modelling the behaviour of concrete.

Several authors; Raynaud *et al*. (2002), Jarny *et al*. (2005) and Huang (2000) were interested in the phenomena related to suspensions rheology.

Further rheological characterizations works were realized on the cement paste, recently (Vikan and Justnes,

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2007). But, with regard to the raw cement, there hasn't been, to our knowledge of study.

For limestone, He *et al.* (2006) was interested in the influences of the solids concentration, of the particles size and the temperature. Their results were indicated that when the concentration in solids increases by 60%, the rheological behaviour is transformed of a slightly dilatant characteristic to a pseudo-plastic characteristic.

Several rheological models were tested to describe rheograms of these suspensions (Vikan and Justnes, 2007):

Power law model:

$$\tau = K\dot{\gamma}^n \quad (4)$$

Bingham plastic

$$\tau = \tau_0 + \mu_p \dot{\gamma}^n \quad (5)$$

Herschel-Bulkley

$$\tau = \tau_0 + K\dot{\gamma}^n \quad (6)$$

Robertson Stiff

$$\tau = A\left(\dot{\gamma} + B\right)^n \quad (7)$$

Casson

$$\tau^{\frac{1}{2}} = \tau_0^{\frac{1}{2}} + \left(\mu_p \dot{\gamma}\right)^{\frac{1}{2}} \quad (8)$$

Sisko

$$\tau = \mu_\infty \dot{\gamma} + K\dot{\gamma}^n \quad (9)$$

Dekee

$$\tau = \tau_0 + \mu_p \dot{\gamma} e^{-\alpha\dot{\gamma}} \quad (10)$$

Where:
τ = Shear stress [Pa]
τ_0 = Yield stress
γ = Shear rate [sec^{-1}]
K = Consistency index
n = Power index (deviation from Newtonian)
μ_p = Plastic viscosity [Pa.s]
μ_8 = Viscosity at infinite shear rate [Pa.s]
α = T dependent parameter, A and B constants

In the study of rheological characterisation Turian *et al.* (1997) showed that the model of Sisko is best adapted to describe the rheological behaviour of suspensions of the silica and gypsum. On the other hand, Yahia and Khayat (2003) was noted that none of these models is in accordance with the obtained rheograms for cement materials, they proposed a model where they integrate a parameter time.

The flow of the suspensions such as the raw material of cement (called the raw) or of the limestone, involves complex interactions between particles. At rest, these particles are organized in structure which gives them a behaviour of solid and when these particles are sufficiently disrupted the suspension has a fluid behaviour. The yield stress which results, is associated with a minimal potential of interaction between the solid particles at rest, influenced by the size of the particles and by the concentration.

In this research, the analysis of the experimental data allowed the identification of parameters, in particular the yield stress value. The objective of this work was to study the effects of the solids concentration as well as the particles fineness on the rheological behaviour of the raw meal suspensions. The use of the Herschel-Bulkley model, permitted to extrapolate from the flow curves the values of yield stress for a rate of shearing zero. Besides, the different rheograms gotten for the suspensions of raw meal have been compared to those of limestone.

MATERIALS AND METHODS

The used materials are mixtures containing 84.74% mass of limestone to which is added a proportions of clay (14.48%), iron ore (0.39%) and sand (0.4%). Thus obtained, these mixtures are called the raw of cement or raw meal of cement and which constitute the raw materials in the industry of the manufacture of cement. The chemical composition of this mixture has been provided by the cement company of Algiers (Table 1).

Eight samples (four for limestone and four for the raw) with a different fineness are obtained by milling of materials provided by the Algerian cement-manufacturer by using a steel cylindrical ball mill. Samples of 10 mL recovered of the ball mill were analyzed using a laser granulometer, the Malvern Mastersizer, 2000. This last is an apparatus, developed mainly during the 70 to 90's and whose operation is based on the theory of the Fraunhofer diffraction according to which the diffraction angle is inversely proportional to the size of the particles (Allen, 1992). Laser granulometry allows the measurement of size

Table 1: Chemical analysis of composition of the raw meal

Compound	CaO	SiO_2	Al_2O_3	Fe_2O_3	Others
Mass (%)	74.02	11.86	7.52	5.27	1.32

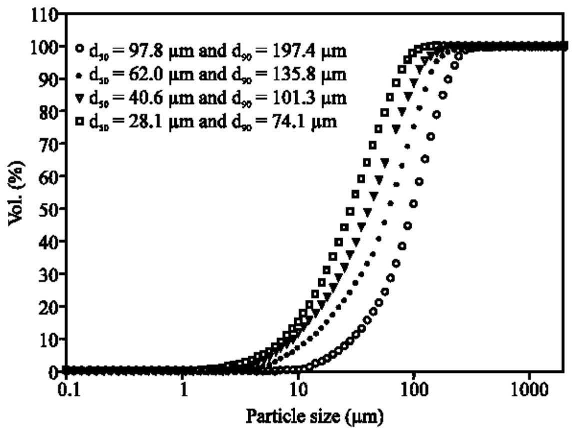

Fig. 1: Particle size distributions of four limestone sample

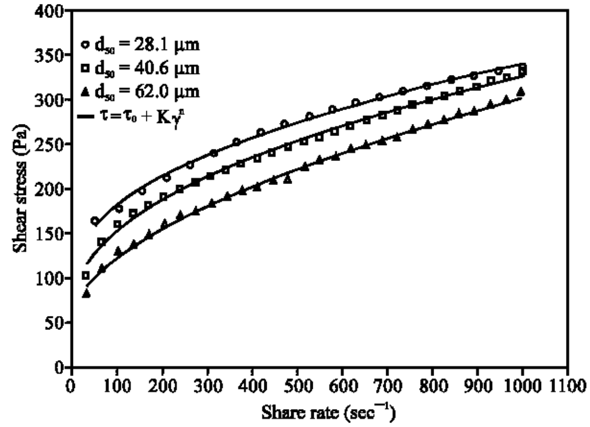

Fig. 3: Flower curve for limestone (ϕ_v = 0.7)

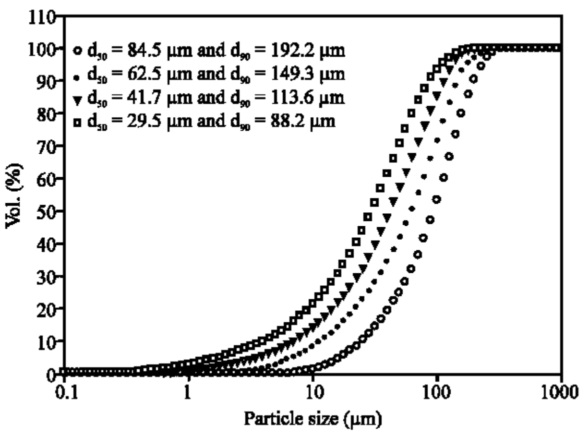

Fig. 2: Particle size distributions of four raw material sample

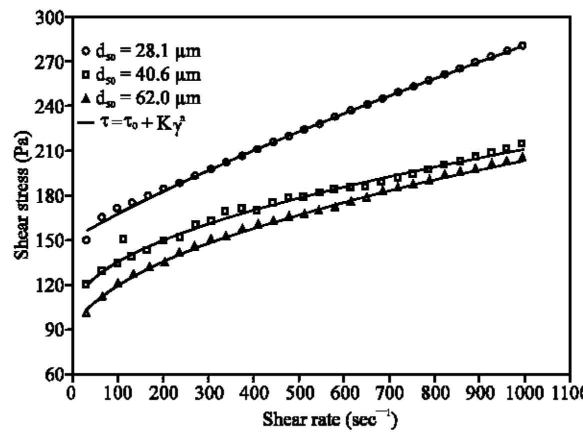

Fig. 4: Flower curve of raw meal (ϕ_v = 0.7)

ranging between 0.05 and 1000 µm, it is thus appropriate, particularly for the materials suspensions which interest this study where the fine particles do not exceed 300 µm.

All the mixtures are carried out under the same conditions. A homogenization of the various components is carried out with a mixer in order to obtain a better particles distribution.

All experiments were conducted in the laboratory of chemical engineering LGC ENCIASET of Toulouse. The granulometric distributions of limestone and the raw are given in the Fig. 1 and 2.

The samples of predetermined granulometry, are then proportioned at various volumes of water 30, 40 and 50%.

The tests rheometric are achieved with a rheometer, AR 2000 of MT instruments equipped with a cone and plate with a diameter 6 cm and an angle of 2°.

This geometry has the advantage of providing a homogeneous shear stress on the sample. The measurements are carried out at imposed shear rate ranging from 0 to 1000 sec^{-1}.

RESULTS AND DISCUSSION

Among the models of rheological behaviour which seem best to represent stationary flow such suspensions, the model of Hersche-Bulkley appears most appropriate to follow the experimental curves.

Figure 3 and 4 provide some examples of rheograms obtained and their approximation by the model of Herschel-Bulkley. In these rheograms, the variable selected as characteristic dimension of the fineness of the particles is the average diameter d_{50}.

The studied flow model has confirmed the behaviour pseudo-plastic of limestone suspensions.

Furthermore, additions (clay, iron ore and sand) forming the raw material of cement did not modify this behaviour, all the samples of the raw paste seem to have a pseudo plastic behaviour. However, an important difference between the limestone and the raw appeared on the level of the parameters of the model.

The comparison of the results obtained on the raw paste with those obtained on the limestone constitute indications on the interaction of the phases constitutive of the dispersed suspensions, such as raw material for cement. In addition, different values have led to parametric analysis of yield stress.

Influence of concentration: In the range of studied concentration, the volumetric fraction of solids does not affect nature of rheological behaviour, which remains rheofluidifiant, It is however very significative on the parameters since, the various concentration ranges could lead to different types of rheograms (Fig. 5, 6), the characteristic shear-thinning is shown in the Fig. 7 and 8 and therefore, the parameters of studied model, in particular the yield stress which is widely affected. Figure 9 and 10 show the influence of the concentration on the yield stress.

It is clearly shown, the yield stress tends to increase with the volumetric fraction of solids. This phenomenon can be explained by the nature of the suspension. Indeed, in a diluted paste, the interparticle distance is so large that the particles are not any more subjected to attractive forces between particles.

The evolution of the yield stress, according to the volumetric fraction of solids can be described by an exponential law:

$$\tau_0 = a e^{b\phi_v} \tag{11}$$

where, a and b are two parameters which can be obtained (Table 2) and which depend on the size and the shape of the particles.

Also, the degree of plasticity increases with the solids concentration (Table 3). These phenomena have been already observed for other materials paste such as coal and quartz (Tangsathitkulchai and Austin, 1988), limestone (He *et al*., 2006).

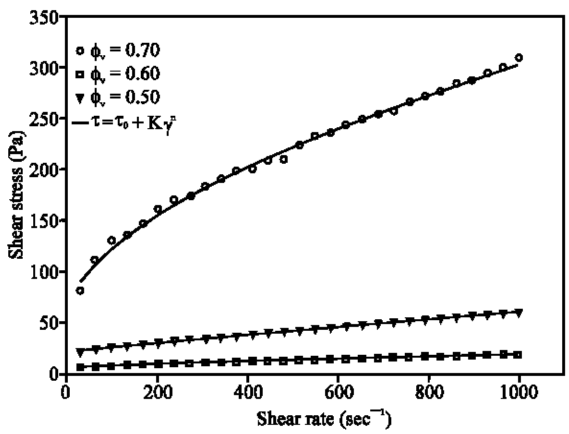

Fig. 5: Effect of solids concentration on the flow curve of limestone pastes (d_{50} = 62 μm)

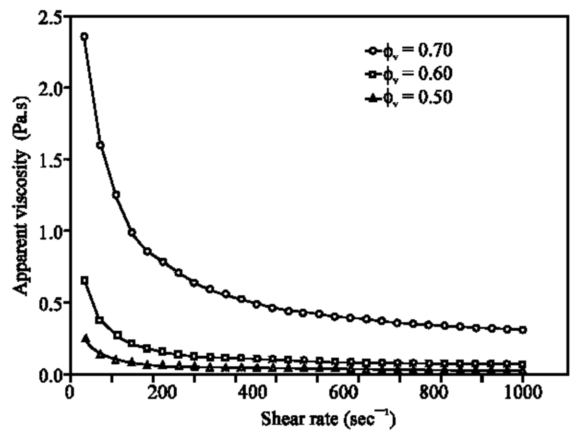

Fig. 7: Effect on solids concentrations on the apparent viscosity of limestone paste (d_{50} = 62 μm)

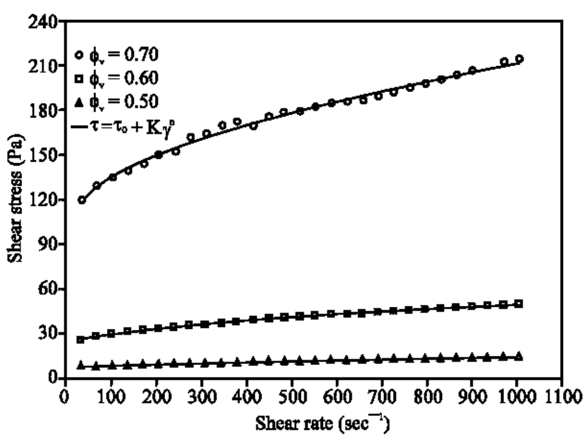

Fig. 6: Effect of solids concentration on the flow curve of the raw pastes (d_{50} = 65.5 μm)

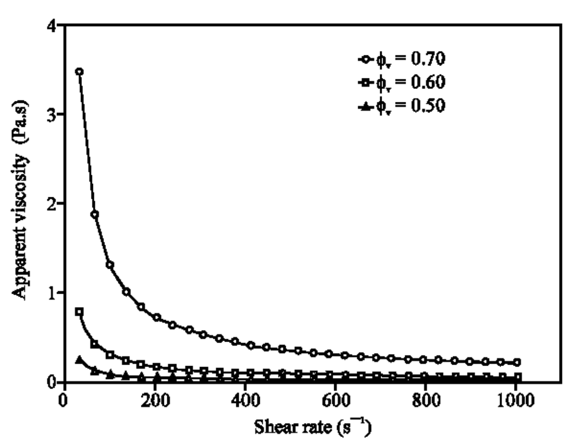

Fig. 8: Effect on solids concentrations on the apparent viscosity of the raw meal paste (d_{50} = 62 μm)

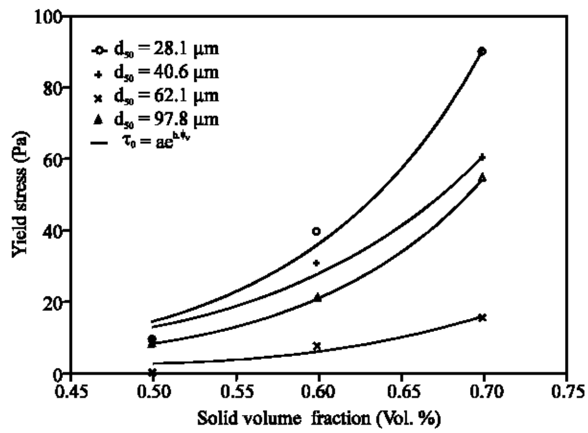

Fig. 9: Effect of solids concentration limestone on the yield stress

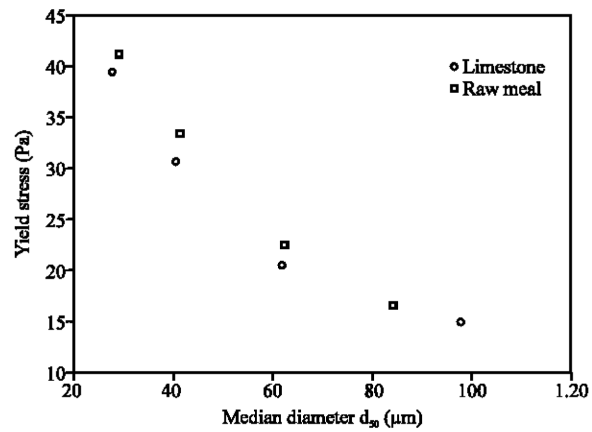

Fig. 11: Effect of granular size on yield stress at $\phi_v = 0.6$

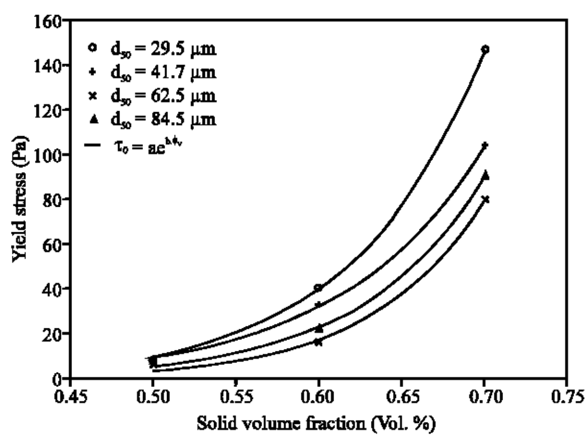

Fig. 10: Effect of solids concentration limestone of raw meal on the yield stress

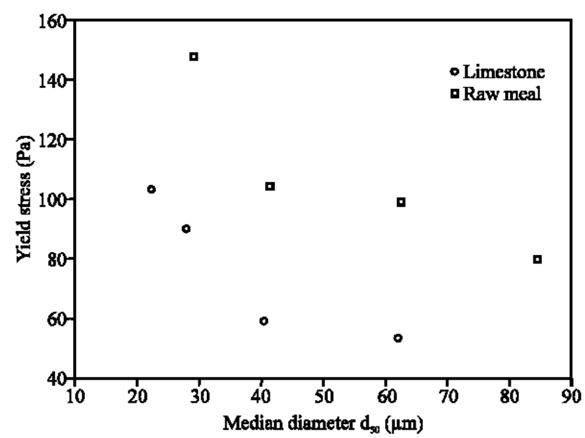

Fig. 12: Effect of granular size on yield stress at $\phi_v = 0.70$

Table 2: Parameter values obtained by fitting experimental data into Eq. 11

Parameters	d_{50} [μm]	a [Pa]	b
Limestone paste	28.1	0.13	9.40
	40.6	0.24	7.85
	62.0	0.06	9.71
	97.8	0.01	9.84
Raw meal paste	29.5	0.016	13.01
	41.7	0.03	11.72
	62.5	0.007	13.50
	84.5	0.002	14.90

Granilometry effects: It is obvious that the rheological properties are not only affected by the concentration in solids, but also by the fineness of the constituent particles.

Some authors have been interested in the study of the role of the fine particles on the rheology of the concentrated suspensions. Tangsathitkulchai and Austin (1988) have indicated that the rheology of concentrated

Table 3: Some values of n power index

Solids volume fraction (Vol. %)	Limestone paste (d_{50} = 40.61 μm)	Raw meal paste (d_{50} = 41.7 μm)
0.70	0.46	0.53
0.50	0.95	0.88

slurries is strongly influenced by the particle dimension (Yue and Klein, 2004), by studying the rheology of quartz pastes have showed that the yield stress decreases with the increase in the size of the particles.

The yield stress can be associated with the physicochemical potential, of attractive type of the particles. It is thus logical that this parameter increases when the size of the particles decreases.

Compared to the limestone, it appears for the same volumetric concentration of solids, the yield stresses obtained on the raw pastes are more higher. However, the deviation between the two quantities increases with the solid volume fraction (Fig. 11, 12).

CONCLUSION

The rheometric tests were carried out on both limestone and raw cement, it has been showed that their rheological behaviour depend on their concentration and their fineness as well. The Herschel-Bulkley's model was checked, it was shown that the rheological behaviour of the raw material is greatly influenced by the presence of limestone particles. The values of yield stress of raw cement were more important than limestone values, it can be explained by the presence of clay. The yield stress decreases with dilution according to an exponential function. This phenomenon is observed for both raw cement and limestone materials. Also, the use of fine particles gives a great yield stress value for each material, which require more water in order to ensure fluidity.

These results contribute to the identification of the raw cement rheology and can be used in the design calculus of milling devices, transport and storage in the manufacturing humid and semi humid cement process.

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