DOI: 10.21276/sjet.2016.4.9.4

Scholars Journal of Engineering and Technology (SJET) **ISSN 2321-435X (Online)**
Sch. J. Eng. Tech., 2016; 4(9):403-411 **ISSN 2347-9523 (Print)**

(An International Publisher for Academic and Scientific Resources)
www.saspublisher.com

Original Research Article

Effect of Sodium Bentonite Clays on the Behaviours of Fresh Sprayed Mortars

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Abstract: This paper presents an experimental study of mortars proportioned with various contents of bentonite. In comparable with a reference formulation, the effect of a bentonite-clay additive is investigated. To determine the adhesive properties the probe tack test was used. The tack test results have been exploited to identify the adhesion strength, the cohesion strength and the adherence force. It is found that the cohesion component monotonically increases with bentonite content. The behaviour of the adherence force is more complex, while the evolution of the adhesive force versus bentonite content displays optima depending upon the tack velocity. The rheological behaviour of the mortars was also considered. The comparison between adhesion tests and rheological measurements has shown that the cohesion force can be related to the yield stress identified on flow curves.
Keywords: Bentonite clays; Rheological measurement; Pull-off test; Fresh mortar

INTRODUCTION

In order to avoid phase separation in cementitious materials, including self-compacting concretes, self-levelling mortars, adhesive mortars, etc., thickening agents are often appealed. In general, these admixtures consist of fine mineral powders (silica fume, metakaolin, etc.), water-soluble polymers (cellulose-ether, starch-ether, etc.) or redispersible polymers (latexes, etc.). Their main action is to fix water in the material, increasing thereby the viscosity of the aqueous solution, which leads to the stabilisation of the suspension [6].

Numerous studies have been devoted to the influence of fine mineral additives on the rheological behaviour of cementitious materials [4, 9]. It has been reported that fine additives, such as limestone, can lead to the decrease of the yield stress and plastic viscosity [9], while ultrafine additives [3] increase water demand leading to the increase of these two rheological parameters. In the present study, the fine additive considered is bentonite clay, which can be considered as an ultrafine (particle size less than a micron). Clay-based admixtures are often used in the formulation of ready-mix mortars in order to improve their placement properties, in particular by pumping. Yet, the influence of clay minerals on the rheological properties of cementitious materials is far to be sufficiently investigated in the literature [10].

In contrast to the rheological behaviour, adhesive properties of cementitious materials in fresh state have been much less considered [5].

The adhesive properties of fresh mortar pastes have been characterized in this study using probe tack tests. This kind of tests has been largely employed to characterize polymer-based adhesives and more recently to investigate the tackiness and various failure modes of smectite muds [1]. Kaci and others [5] have used the probe tack test to characterize the adhesive properties of cementitious materials. They have shown that tack measurements allow dissociating several aspects of practical interest, related to adhesive properties [5]:

-Interface adherence, which expresses the product’s ability to stand on its support.

-Cohesion: this property is related to the yield stress, and characterizes the material’s resistance to flow initiation under extension.

-Adhesion strength: this quantity encompasses both cohesion strength and viscous dissipation, and can be employed to characterize adhesion properties under flow conditions. Kaci and others [5] have investigated the influence of water-soluble polymers on the adhesive properties of fresh mortar joints. For those materials used in practice as thin joints to bind construction blocks together, the aim was to characterize the adhesive properties that guarantee an adhesion to the surfaces but not to the tools. In this investigation we perform an extension of the aforementioned work. In particular, we consider also the case of another type of thickening agent, which is bentonite clay. Mohamed Abdelhaye and others [1] considered the tackiness properties of bentonite suspensions using the probe tack test. It is then interesting to investigate the influence of bentonite on the tackiness of mortars, since this additive is often used in ready-mix mortars.

Finally, in order to complete the characterization of placement properties of mortars, the rheological properties are determined at different bentonite contents and compared to the adhesive properties.

MATERIALS AND EXPERIMENTAL METHODS

Mortar mix-design

The mortar mix design used in this study is given in Table 1.

Table 1: Mortar mix design used in this study.

Constituent	Portland cement	Lime	Siliceous sand	Water	Bentonite clays	Air entraining agent
% by weight of the binder	15	5	80	15	0,05-2	0,01

The binder comprises a Portland cement and a hydraulic lime. The other constituents consist of silica-based sand with controlled granular distribution and an air-entraining admixture (Table 1). The mortar composition corresponds actually to a basic version of commercially-available render mortars.

In order to minimize phase separation, the particle size distribution of sand has been obtained by combining two contrasted granulometries: a fine sand with a mean particle size of 0,41 mm, and a coarse sand with mean particle size of 1,13 mm. The air entraining agent guarantees moderate rheological properties, within the resolution range of our rheometer. The water dosage rate is fixed to 15% by weight for all the investigated pastes. The particle size distribution of sand, determined by sieving, meets the requirements of the standard EN 196-1 and ISO 679-2009.

Table 2: Chemical composition of bentonite, wt%.

LOI*	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	MgO	CaO	Na_2O	H_2O	**Total**
8.75	53.72	19.12	0.85	4.93	3.29	5.28	3.64	0.44	**100.0**

** LOSS on ignition at 1100°C*

A sodium bentonite was usually employed in drilling muds and retaining fluids formulations [8]. Such additives serve as thixotropic and thickening agents, and must present particular rheological properties such as a high yield stress to prevent sedimentation [7]. The bentonite used in this work originates from a bentonite quarry in Milos Island, Greece. The chemical composition of bentonite is shown in Table 2.

The mortars were prepared by the following steps:

(1) Mixing the dry constituents at low speed (60 rpm) for 30s
(2) Adding the required amount of water
(3) Mixing at low speed for 30s
(4) Stopping the mixer for 30s. During this time, the material is manually mixed to recover the sticking material to the container's wall.
(5) Mixing at high speed (125 rpm) for 60s.

Adhesive test

The probe tack tests have been performed on a rheometer of TA Instruments. A fine layer is inserted between two parallel plates of high roughness, which allows minimizing wall slippage. The material is left to rest for 2 minutes after casting, in order to avoid possible memorial effects. The plates are then separated under a constant velocity, which is varied among the following values: [10, 50, 100, 300 and 500] µm/s. Under each imposed pulling velocity, the normal stretching force is measured concurrently with the instantaneous distance between both plates. Knowing the initial weight of the mortar, the measurement of the final weight enables us to determine the amount of material remaining on the mobile plate at the end of the test.

Fig-1. Tack geometries - Upper plate; Bottom plate; A tack test in process

Rheological measurements

The rheological properties are determined with the same rheometer, equipped with the vane geometry. Yet, with this geometry the tested material is not subjected to a uniform shear rate. This condition is usually required in rheological measurements in order to measure actual material properties, and to have an analytical relationship between the measured torque/rotational velocities and shear rate/shear stress. Nevertheless, vane geometry has been retained since it is appropriate for high yield stress fluids such as dense granular suspensions, including mortars [5], as slippage can be avoided and the material can be sheared in volume.

The yield stress is measured with the vane-cylinder geometry in stress controlled mode in which a "ramp" of steps of increasing stress levels is applied to the vane immersed in the material, and the resulting shear rate is measured as a function of applied stress. The yield value is determined from the critical stress at which the material starts to flow. Between two successive steps there is no pre-shear or rest. The measurement point duration is set and assumed that equilibrium reached at each stress condition to obtain a flow curve.

Depending on each specific experiment, we have to perform the test at least three times to determine the best possible procedure. In the first run, the interval between two successive steps must be chosen large enough to reduce the duration of the test. The yield stress is determined, but with a low precision. And then, for the latter runs, the measuring points must be increased around the determined yield point. That would help to determine a high accuracy yield stress of the test sample.

However, in actual experiments, almost all cases, the transition from solid to liquid state is occurred gradually and is hard to detect. Therefore, it is difficult to determine the exact value of the yield stress. So, different models have been developed in order to determine the value of the yield stress as well as other rheological parameters by fitting the flow curves' data with the model's equation. In case of mortars containing a high polymer dosage, a third parameter is necessary to model the flow curves. At equilibrium the shear stress is related to the shear rate is a non-linear way:

$$\tau = \tau_0 + K.\dot{\gamma}^n$$

The consistency coefficient K, the fluidity index n, and the yield stress τ_0 are three parameters characterize Herschel-Buckley fluids. The consistency K is a simple constant of proportionality, while the flow index n measures the degree to which the fluid is shear-thinning or shear-thickening.

In some cases the use of Herschel-Bulkley model leads however to non-physical values of the yield stress (negative), this parameter is then determined by the applied stress at which we obtained a finite shear rate (0.01 s^{-1}). These tests led to the determination of three rheological parameters, including yield stress, consistency coefficient and fluidity index. The influence of various types of admixtures on the shear properties was investigated through these three rheological parameters.

RESULTS AND DISCUSSIONS

Tack test results

Figure 2 illustrates typical time evolutions of the normal force, under varying velocities and for the mixture with 1% bentonite. A semi-logarithmic scale has been retained to bring out the behaviour around the peak. The curves are all qualitatively similar. The initial force increase can be related with elastic and visco-elastic deformations, under mixed conditions of shear and extensional flows. After reaching a peak which gets more pronounced as velocity increases, the normal force decreases abruptly during the paste progressive rupture, and we observe an inward flow towards the plates' center under tension. After completion of the rupture process, a residual force level is reached.

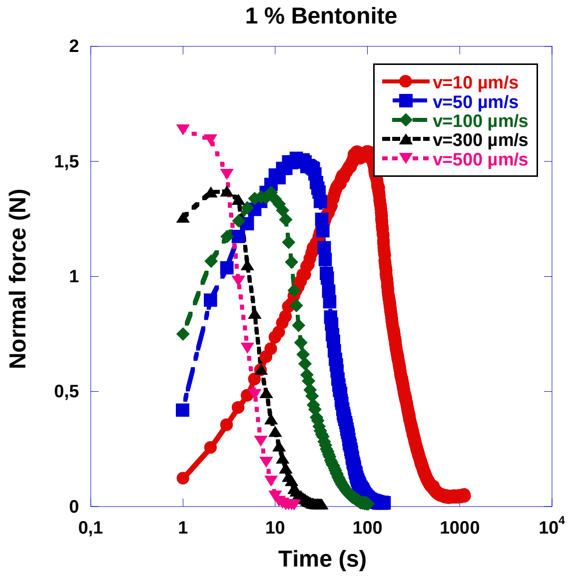


Figure 2: Evolution of pulling force for varying velocities for a mixture with 1% bentonite.

Three relevant properties can be directly identified from tack test curves of Figure 2:

- The peak value F_{max} is employed to determine the *adhesion strength* of the material, originating both from flow resistance (owing to viscous effects) and the material's intrinsic cohesion at rest.
- The *cohesion strength* will therefore be identified by considering the adhesion force for pulling velocities tending to zero, i.e. when no viscous effects are present under quasi-static conditions.
- Finally, the residual force at the end of the pulling test corresponds to the weight of the material still remaining on the mobile plate, and allows characterizing the *adherence* at the plate-mortar interface.

Figure 3 shows that the adhesion force is practically independent of the pulling velocity. However, we can notice a minimum in adhesion around 100 µm/s which still remains to be investigated. The observed results can be related with the fact that bentonite additions increase mostly the yield stress and not the viscosity.

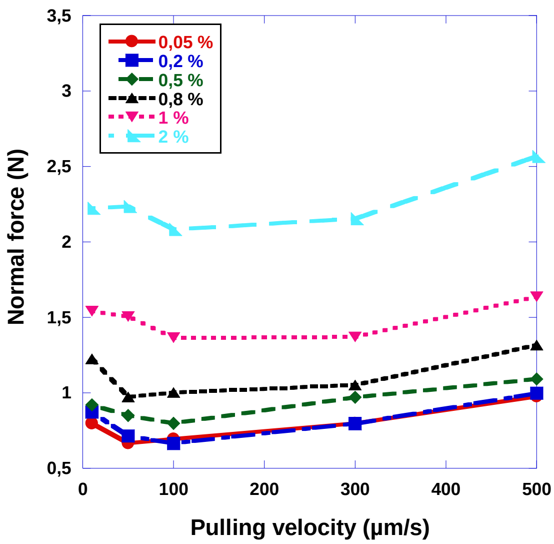


Fig-3: Evolution of the adhesion force as a function of the pulling velocity for varying bentonite contents.

DOI: 10.21276/sjet.2016.4.9.4

Figure 4 displays the evolution of the cohesion force with bentonite content. A significant increase can be noticed for contents higher than 0.5 %, which can be correlated to the shear yield stress.

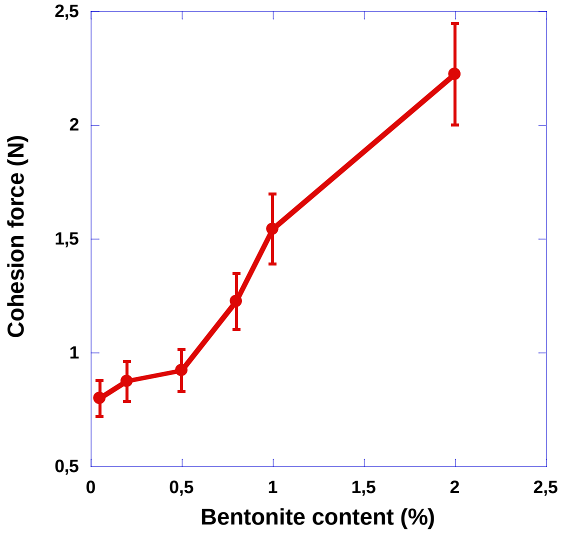


Fig-4*:* **Evolution of the cohesion force with bentonite content.**

The evolution of the adherence force is illustrated in Figure 5. The observed behaviour is unexpectedly complex. For each given velocity, the adherence force decreases at first when bentonite is added to the mixture. A minimum in adherence is reached at 0,5%, then increases to a maximum value at 1%, and decreases afterwards. To understand this result, we have to search for possible modifications in rupture behaviour during the tack test, depending on the bentonite content. In a general way, if the product displays an important cohesion, this may imply a decrease in interface adherence. The latter assumption is fulfilled for low and high bentonite contents, but not for intermediate values (between 0,5% and 1%), that are characterized by an increase of both cohesion and interface adherence. Finally, for low pulling velocities, a good adherence has been observed (Figure 6) and adherence decreases abruptly for higher velocities.

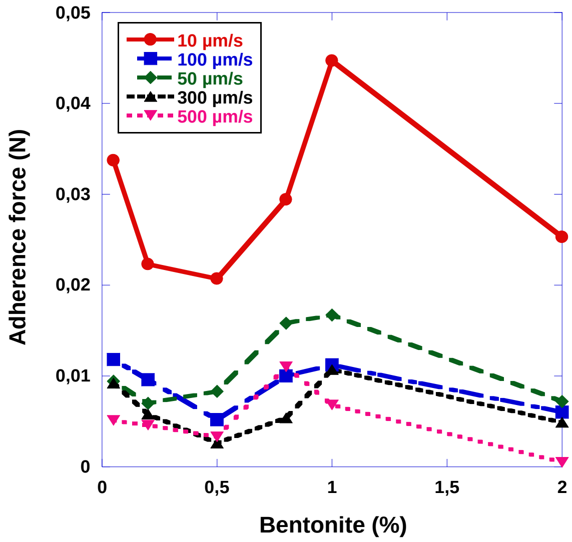


Fig-5: **Evolution of the adherence force versus bentonite content for different pulling velocities.**

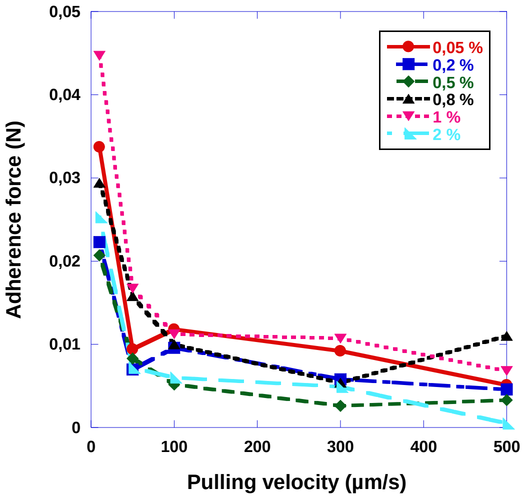
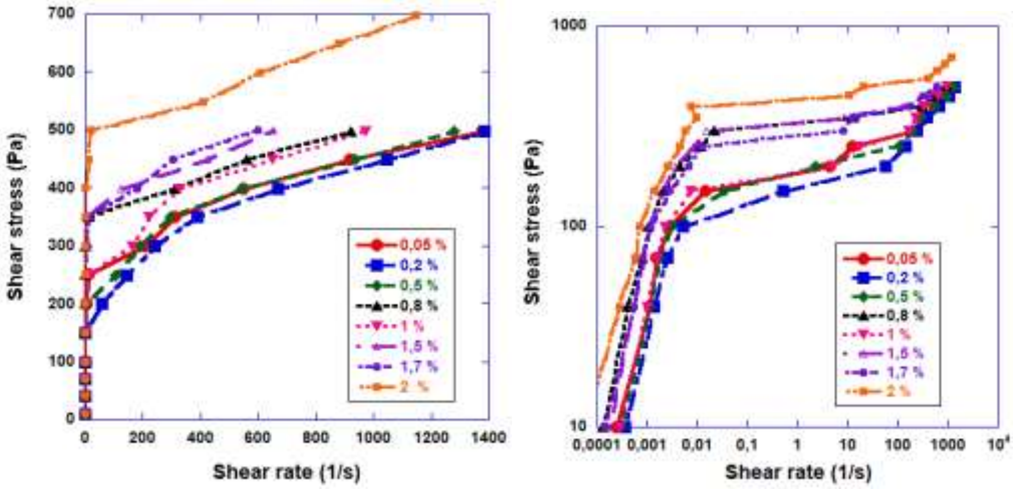


Fig-6: **Evolution of the adherence force with pulling velocity for varying bentonite contents.**

Rheological measurements

The flow curves obtained under shear stress controlled mode are displayed in figure 7 for varying bentonite contents, in both linear and logarithmic scales in order to highlight the evolution of shear stress versus shear rate at low shear rates. We can see in the linear plots that there are two qualitatively different rheological behaviors depending of the bentonite content: shear-thinning behavior at low contents of bentonite and Bingham fluid at high bentonite contents. To see this more clearly, the flow curves are zoomed in at the low shear rates in figure 8. At low content of bentonite, the flow curve is that of a shear-thinning fluid with a yield stress (Herschel-Bulkley fluid). As it can be clearly seen, at high bentonite content, e.g. 0.8 %, after the applied stress exceed the yield value, the relationship between the shear stress and shear rate is almost linear with a low slope. It can be considered that the viscosity is constant and the mortar behaves as a Bingham fluid. We observe the same behavior at 1; 1,5; 1,7 and 2%.



(a) **(b)**

Fig-7: Flow curves obtained in rheological measurements of mortars in formulation with bentonite: (a) Linear scale; (b) Logarithmic presentation

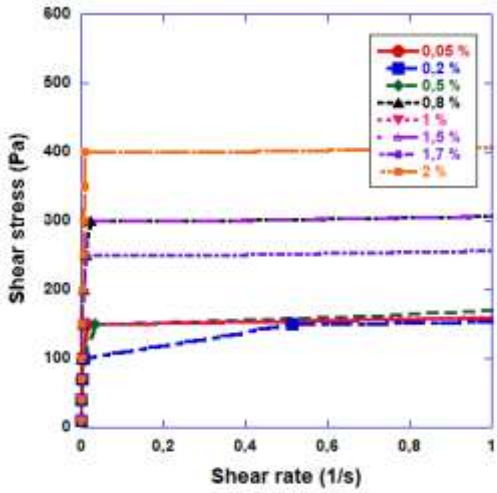


Fig-8: Rheological flow curves in low shear rate

The evolution of the yield stress is shown in Figure 9. It indicates that increasing the bentonite concentration first decreases the yield stress. A minimum is observed at 0,2 %. It is to be noted that this minimum is not an artifact since the test has been repeated three times. This is followed by a significantly increase of the yield stress with bentonite concentration. We see that the yield stress and the cohesion force evolutions with bentonite content are fairly similar.

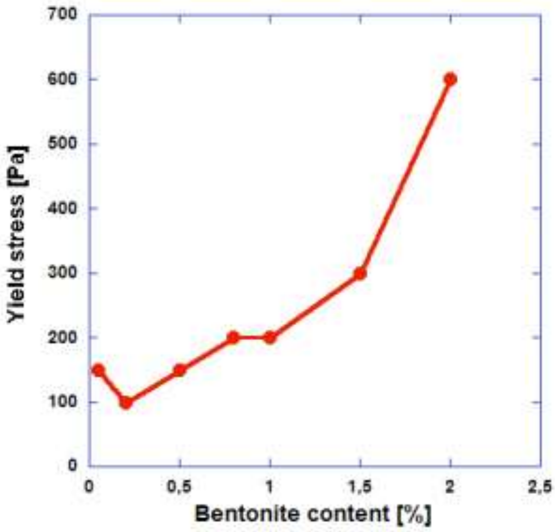


Fig- 9: Evolution of the yield stress with bentonite content

Two other rheological parameters, including the consistency and the fluidity index, were determined by performing the best fit with the Herschel-Bulkley model. The evolutions of consistency coefficient and fluidity index versus bentonite concentration are represented in Figure 10. The consistency of the mortars decreases with the increasing bentonite content. This decreasing is monotonic and reflects the decrease of the viscous drag effects with bentonite content.

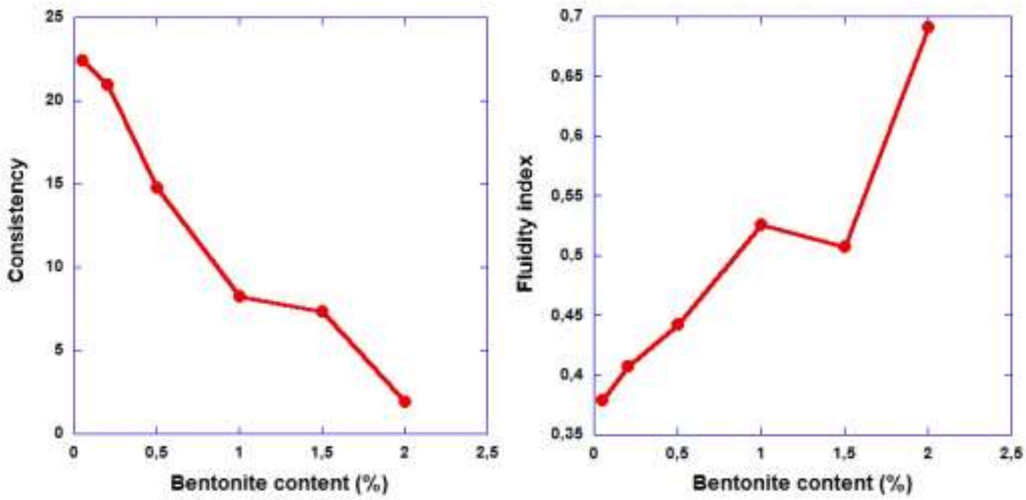


Fig-10: Evolution of consistency and fluidity index with bentonite contents

The fluidity index first increases with the increasing of bentonite content. We observed a maximum at 1%, followed by a minimum at 1,5 %. However the gap between these two quantities is small. Overall, we can see that we have increase of the fluidity index of the mortar with bentonite content. This evolution means that the mortar becomes less shear thinning at higher bentonite content. At 2%, the value of fluidity index approach 1, at which the mortar behaves like a Bingham elasto-plastic fluid.

CONCLUSIONS

In this paper, we have undertaken a systematic study of the properties of fresh mortars that can be derived from tack tests and rheological experiments. Starting with a reference mortar paste, the effect of bentonite additive has been investigated.

The peak normal force, the cohesion force and the yield stress all monotonically increase with bentonite content, while the adherence force and viscosity's evolutions with bentonite content are more complex. Several mechanisms have been proposed to interpret the observed results; in particular the role of the viscosity of the fluid phase should be investigated further by performing viscosity measurements for the suspending fluid alone.

The comparison between adhesion tests and rheological measurements has shown that the cohesion force can be related to the yield stress identified on flow curves, while the viscosity correlates well with the interface adherence.

Finally, as a perspective, owing to the complex mechanical conditions prevailing during adhesion tests and rheological measurements, numerical simulations should be performed in large deformation analysis, in order to gain further insight into the physical processes involved.

Acknowledgements: We are grateful to Vinh University for sponsoring this investigation.

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