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Abstract.

One industrial process used by Nexter Munitions to manufacture pyrotechnical materials consists in preparing an emulsion of wax in TNT (2,4,6-trinitrotoluene) and adding Aluminium and ONTA (3-nitro-1,2,4-triazole-5-one) particles. When the suspension is homogeneous, it is allowed to flow by gravity through a pipe located at the bottom of the tank and to fill up a shell body. The suspension is characterized by a solid volume fraction of 53.4%, which leads to high viscosities. In some circumstances, the emptying time is prohibitively long and the economic profitability is reduced. This study has been performed to make the emptying time lower with the constraint of unchanged volume fractions and grains mean diameter. So, we investigated the influence of the grain size distribution on the suspension viscosity. Different samples of Aluminium and ONTA have been used, with rather small differences in grain size distributions. The suspensions have been prepared in the industrial tank and the flow cast times measured. It has been observed that they differ by one order of magnitude. To avoid situations with too high emptying times, a procedure has been implemented to make prior characterization of the suspension rheology. Because of particles sedimentation and emulsion destabilisation, the classical Couette rheometer is not adapted. So, we designed and built a small size tank (113 cm³), where the suspension is continuously stirred and kept homogeneous. The measurement of the torque and rotational speed together with the use of the Couette analogy allowed us to observe an Ostwald fluid behaviour (flow consistency index k, flow behaviour index n). To gain in prediction, we established a correlation between the measured (k, n) values and the grain size distributions. We characterized each suspension by the ratio of ϕ to ϕ_m, where ϕ is the solid volume fraction (imposed by the commercial specifications) and ϕ_m is the maximum packing fraction. Because of the strong analogy between concrete and energetic paste, we chose the widely used De Larrard model to compute ϕ_m. A linear dependance between the ratio ϕ/ϕ_m and the indices k and n was observed. The second step was to provide an analytical expression for the flow cast time of a power-law suspension from a tank with a given geometry. Considering the large difference between the industrial inner tank diameter and the evacuation pipe diameter, we assumed that all the pressure drop was located in the evacuation pipe. Then, extending the Hagen-Poiseuille equation to Ostwald fluid, we were able to predict the emptying time with the knowledge of k and n. Experimental and predicted emptying time are in very good agreement. This work helped the industrial manufacturer to divide the emptying time by a factor 12.

Keywords: Concentrated suspension ; explosive materials ; Couette analogy ; Ostwald fluid ; maximum packing density ; De Larrard model ; Hagen-Poiseuille equation.

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Theory

Modified Hagen-Poiseuille Equation

The Hagen-Poiseuille formula predicts the flow rate through a cylindrical tube for a given driving force (imposed pressure difference and/or gravitational volumetric force). The industrial tank, used to prepare XF13333 formulations, can be represented in a simplified way by Figure 1 where $\vec{g}$ is the acceleration due to gravity (9.81m.s^{-2}), R' the internal radius of the tank (m), R internal radius of the outlet pipe (m), P_2, P_1 and P_0 (Pa) the pressures at the respective heights H_i, H_f and H_0 (m), $H(t)$ represents the height at time t (s) and h the height between H_f and H_0.
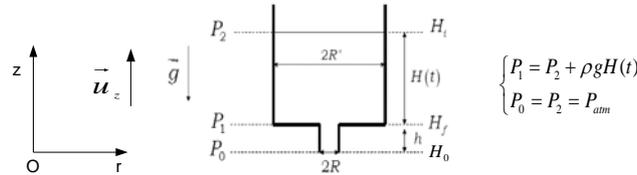

Figure 1: Schematic representation of the industrial tank.

From the general Navier-Stokes equation and in the configuration described by Figure 1, the viscous stress tensor can be written, in flow direction (for a projection on the axis O_z) and after integration following *r*:

$$\sigma'_{rz} = \frac{r}{2}\left[\frac{\partial P}{\partial z} - \rho g z\right]$$

Eq. (1)

For a power law type fluid, the viscous stress tensor can be written as:

$$\sigma'_{rz} = k\left(\frac{\partial v_z}{\partial r}\right)^n$$

Eq. (2)

where k (Pa.s^n) is the flow consistency index, *n* the flow behaviour index and v_z the flow velocity along the axis O_z. Finally, after some simplifications, the volumetric flow rate q (m³/s) of an Ostwald fluid in the configuration described by Figure 1 is given by the modified Hagen-Poiseuille equation:

$$q = \frac{\pi n}{1+3n}\left(\frac{1}{2k}\right)^{1/n}\left[\rho g\left(1 + \frac{H(t)}{h}\right)\right]^{1/n} R^{\frac{1+3n}{n}}$$

Eq. (3)

With Eq.(3), the emptying time *t* can be calculated.

Maximum Packing Density Calculation

The emptying time *t* of XF13333 suspensions is principally governed by the maximum packing density ϕ_m [1]. To calculate this parameter, the De Larrard model [3] is used because it appears to be the most adaptable to evaluate the compacity of our solid binary mixtures. For more details on the De Larrard model, interested readers can refer to his book [4].

Experimental Set-up, materials and protocol

To determine the correlation between the Ostwald parameters, *k*, *n*, and the maximum packing density ϕ_m, five XF13333 suspensions, with different ϕ_m and same ϕ (equal to 53.4%), are characterized using the unconventional "RheoXF" rheometer (see [5] for details and experimental protocol).

To compare experimental and theoretical emptying times, nine XF13333 formulations, with different ϕ_m and same ϕ, are elaborated in the industrial tank. The time *t* required for 5700 cm³ of suspension to flow by gravity from the tank down to a shell body is measured [5].

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Results

Correlation Between The Flow Consistency Index, The Flow Behaviour Index And The Maximum Packing Density

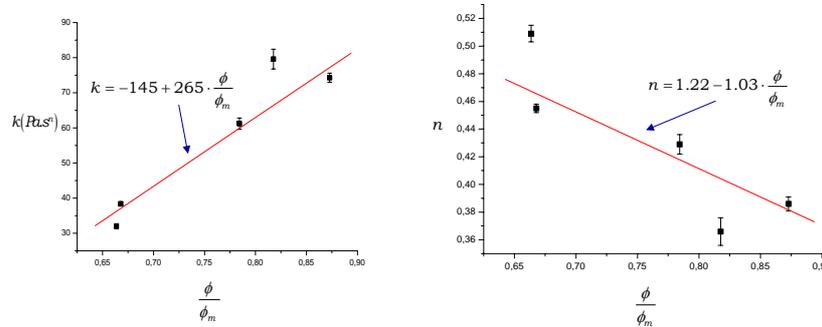From the n and k experimental values for each ratio ϕ/ϕ_m, two linear correlations can be established.

$$k = -145 + 265 \cdot \frac{\phi}{\phi_m}$$

$$n = 1.22 - 1.03 \cdot \frac{\phi}{\phi_m}$$

Figure 2. Correlation between k, n and ϕ_m ϕ/ϕ_m

Correlation Between The Flow Consistency Index, The Flow Behaviour Index And The Maximum Packing Density

To calculate the theoretical flow time t, the modified Hagen-Poiseuille equation (Eq.(3)) is used. As shown on Figure, an acceptable agreement is observed between experimental and theoretical results.
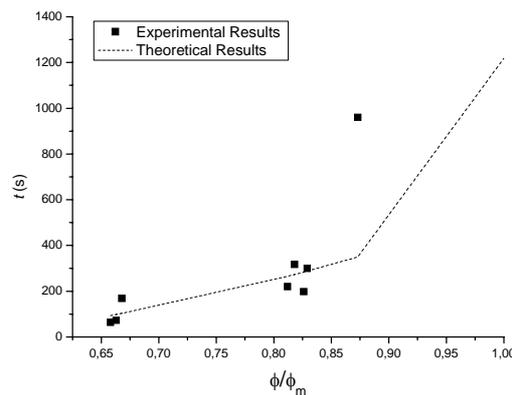

Figure 3. Correlation between the experimental flow time and the theoretical flow time calculated from the modified Hagen-Poiseuille equation.

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