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1 **USE OF RAM EXTRUDER AS A COMBINED RHEO-TRIBOMETER TO STUDY**  
2 **THE BEHAVIOUR OF HIGH YIELD STRESS FLUIDS AT LOW STRAIN RATE**

3

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14

15 **Abstract:**

16 We propose in this work to provide an efficient and simple extruder device able to evaluate  
17 the rheological and tribological behaviour of high yield stress fluids, such as extrudible  
18 materials. An extruder able to measure simultaneously both the friction force acting on the  
19 extruder wall and the total extrusion force is developed. Based on previous studies, an  
20 efficient and accurate method of data analysis is then proposed and applied in order to obtain  
21 both a flow curve and a tribological law. Experimental tests are performed on soft modelling  
22 clay, kaolin paste and cement-based materials. Results are compared to conventional  
23 rheometry measurements. This comparison helps to evaluate the accuracy of the proposed  
24 experimental device and procedure.

25

26 **Keywords:** Extrusion, rheology, tribology, yield stress.

27 **Nomenclature**

28 d: Die orifice diameter (m)

29 D: Extruder barrel diameter (m)

30 F: Total extrusion force recorded at the ram (N)

31  $F_{fr}$ : Friction force on the extruder barrel (N)

32  $F_{pl}$ : Shaping force required to ensure the flow in the die land (N)

33  $F_{pl1}$ : Contribution of  $F_{pl}$  induced by the bulk deformation in the shaping zone (N)

34  $F_{pl2}$ : Contribution of  $F_{pl}$  induced by the friction acting along the dead zone surface (N)

35  $F_{pl3}$ : Contribution of  $F_{pl}$  induced by the friction along the exit orifice surface (N)

36  $h_c$ : Height of the cylinder tool (m)

37  $h_v$ : Height of the vane tool (m)

38  $k_{fr}$ : Apparent friction factor ( $\text{Pa} \cdot (\text{s} \cdot \text{m}^{-1})^{n_{fr}}$ )

39  $k_s$ : Shear consistency -Herschel-Bulkley behaviour ( $\text{Pa} \cdot \text{s}^n$ )

40  $K_w$ : Wall friction stress (Pa)

41  $K_{w0}$ : Wall friction yield stress (Pa)

42  $L_B$ : Length of remaining billet (m)

43  $L_{dz}$ : Static or dead zone length (m)

44  $L_O$ : Exit orifice length (m)

45 m: Ratio of the wall friction stress to the bulk shear stress

46 M: Maximum torque recorded during vane tests (N.m)

47  $n_{fr}$ : Friction index

48  $n_s$ : Shear flow index -Herschel-Bulkley behaviour

49  $R_c$ : Radius of the cylinder (m)

50  $R_t$ : Valley to peak value of the surface roughness profile (m)

51  $R_v$ : Radius of the vane tool (m)

- 52  $V$ : Ram velocity ( $\text{m}\cdot\text{s}^{-1}$ )
- 53  $V_i$ : Velocity at the interface between extruder wall and material billet ( $\text{m}\cdot\text{s}^{-1}$ )
- 54  $\dot{\gamma}$ : Shear rate ( $\text{s}^{-1}$ )
- 55  $\theta$ : Conical die entry defined as half the included angle of the dead zone
- 56  $\sigma_0$ : Bulk elongational yield stress (Pa)
- 57  $\tau$ : Bulk shear stress (Pa)
- 58  $\tau_0$ : Bulk shear yield stress (Pa)

59

## 60 **1. Introduction**

61

62 Extrusion is a highly efficient forming process used in a wide range of industries such as food  
63 engineering, ceramics, pharmaceuticals, building materials, etc. The extrusion flow properties  
64 are directly linked to the material rheology and tribology. An accurate evaluation of the  
65 rheological and tribological behaviour of the extruded materials is crucial in order to optimize  
66 the process.

67 In Händle's book on ceramics extrusion, ram extrusion is defined as a discontinuous shaping  
68 process in which a plastic material is pushed through a die or matrix by a piston, whereby the  
69 geometry of the die determines the extrudates profiles (Doll et al. 2007). The use of a ram  
70 extruder as a rheometer has already been studied many times (Avitzur 1983; Basterfield et al.  
71 2005; Benbow and Bridgwater 1993; Hill 1950; Horrobin 1999; Mimoune and Aouadja 2004;  
72 Toplak et al. 2007; Li and Bridgwater 2000; Benbow et al. 1991).

73 In this study, we focus on the development and the use of a ram extruder device dedicated to  
74 the study of the rheological and tribological behaviour of high yield stress fluids. The  
75 developed apparatus is able to provide independently the wall shear stress at the  
76 paste/extruder barrel interface and the pressure required in the die land while common

77 capillary rheometer only provides the total pressure drop required to ensure the flow in the die  
78 land. This is of great interest because data analysis of common capillary tests requires  
79 assumptions or tests performed with different die geometry in order to separate the pressure  
80 drop contribution due to the wall friction from the contribution due to plastic deformation in  
81 the shaping area.

82 In a first approach, Benbow and Bridgwater (Benbow and Bridgwater 1993) and Hill (Hill  
83 1950) have used the ram extrusion geometry as a tool providing a comparative parameter (for  
84 example “extrusion pressure” or “bulk yield stress”) which is directly linked to the die  
85 pressure drop. These studies have contributed to improve the ideal work equation used for  
86 plastic materials. The effect of ram velocity can also be studied with such data analysis  
87 methods (Benbow and Bridgwater 1993).

88 Those methods only provide comparative parameters which depend on the ram extruder  
89 geometry as shown by recent studies (Basterfield et al. 2005; Toplak et al. 2007). For  
90 example, the measured values of extensional yield stress can be far larger than the extensional  
91 yield stress of the material computed from rotational rheometry (Srinivasan et al. 1999; Zhou  
92 and Li 2005a). However, those methods are the most widely used with a ram extruder and  
93 have been used in several studies to analyse the extrudability of materials or to evaluate the  
94 material’s flow behaviour (Alfani and Guerrini 2005; Cheyne et al. 2005; Martin et al. 2006;  
95 Mascia et al. 2006; Rahman et al. 2002; Srinivasan et al. 1999; Zhou and Li 2005a; Zhou and  
96 Li 2005b).

97 For plastic material and using plastic theory and slip lines analysis, several studies provide  
98 more accurate modelling of the force required to overcome the diameter variation using the  
99 material elongational yield stress. Those methods require assumptions on the velocity field,  
100 slip line design and limit conditions. This was attempted by Horrobin (Horrobin 1999;

101 Horrobin and Nedderman 1998), Avitzur (Avitzur 1983) or Kobayashi and Thomsen  
102 (Kobayashi and Thomsen 1963).

103 In recent studies, theoretical frames have been developed to compute intrinsic rheological  
104 parameters such as yield stress, consistency or flow index from extrusion tests.

105 For example, Basterfield et al. (Basterfield et al. 2005) proposed an adaptation of the Gibson  
106 equations (Gibson 1988) which is used to compute the viscosity and flow index of a power  
107 law fluid from orifice extrusion force data. The authors assume a radially convergent flow  
108 from the extruder body to the exit die. The proposed model assumed the converging flow to  
109 be purely elongational.

110 Toplak et al. (Toplak et al. 2007) have recently developed a new method to compute the yield  
111 stress of a material from its gravity draining through an orifice. The authors assume the flow  
112 to be similar to the Poiseuille flow through a cylindrical barrel with a diameter equal to the  
113 exit die one. The proposed model assumes a shear flow and was successfully applied to wide  
114 bore capillary rheometer with small die cross sections.

115 Extrusion is also greatly influenced by the material tribology as shown in several studies  
116 (Estellé et al. 2006; Mankar et al. 2004; Perrot et al. 2006; Toutou et al. 2005). Friction may  
117 occur in the die land area (Benbow and Bridgwater 1993), but also between the extruder  
118 barrel and the material billet as shown in (Mankar et al. 2004; Perrot et al. 2006). Moreover,  
119 for high solid volume content materials such as cement-based material, friction forces are the  
120 the most important resistant force to overcome in order to ensure the extrusion flow. As a  
121 consequence, it is crucial to study what happened far from the die land. This means that the  
122 tribological behaviour of the material has to be determined.

123 This leads to the conclusion that the total extrusion force can be split into two contributions:  
124 the force required to overcome the diameter decrease and the force required to overcome the  
125 friction force acting on the extruder barrel.

126 In this study, we propose to design a novel ram extruder which makes it possible to study both  
127 rheological and tribological behaviour of high yield stress fluids (shear yield stress larger than  
128 1 kPa). The developed apparatus is able to measure simultaneously the friction force between  
129 the extruder and the material billet and the total extrusion force. This extruder is then tested  
130 on three different materials: a cement-based material, a kaolin clay paste and commercial soft  
131 modelling clay. An adaptation of the model developed by Basterfield et al. is done  
132 (Basterfield et al. 2005) to obtain an accurate estimation of the rheological modelling  
133 parameters taking into account the friction acting along the dead zone (which acts as a tapered  
134 die).

## 135 **2. Materials and protocols**

136

### 137 **2.1. Tested materials**

138 Three pasty materials are tested. They exhibit a yield stress ranging from 15 to 50 kPa. The  
139 first one is a commercial soft modelling clay which is known to exhibit a viscoplastic  
140 behaviour with negligible elastic properties after short compression (Estellé et al. 2006). The  
141 second material is a mix of kaolin clay powder and water (water/kaolin mass ratio equal to  
142 38%). The kaolin clay used is a Powdered Polwhite BB from Imerys (Kaolins de Bretagne,  
143 Ploemeur, France). The largest clay grain size is close to 40  $\mu\text{m}$ . The mass mean kaolin grain  
144 size is close to 9  $\mu\text{m}$ . The third material is a cement-based material containing a large amount  
145 of fly ash. The dry powder consists in a mix of 70% of fly ash and 30% of cement. The fly  
146 ash has an optimized grain size distribution (mix between micronised and natural) and the  
147 cement is an Italcementi type I 52.5 R. The liquid phase is composed by tape water (96 % in  
148 mass) and methylcellulose 4% in mass). Such admixture is a polymeric suspension acting as a  
149 viscosity enhancer agent in order to reduce drainage. Tests on cement-based paste are



150 performed without delay after mixing in order to avoid rheological changes due to chemical  
151 effects.

152 In the present study, the paste compressibility has been neglected. This assumption is  
153 supported by two main reasons. Firstly, the study does not concern highly viscous polymer  
154 melts which are very sensitive to pressure (Park et al. 2008; Dealy 1995). Secondly, the  
155 compressibility effects in the die land remain limited because the recorded pressures are  
156 relatively low (under 500 kPa). Such hypothesis seems to be relevant in the present study but  
157 it cannot be held in high pressure capillary flow of firm polymer pastes (Mitsoulis and  
158 Hatzikiriakos 2009; Mitsoulis et al. 2007).

159 The rheological behaviour of the three tested pastes is visco-plastic. It is important to note that  
160 the extruder is efficient only if the tested material remains homogeneous. Thus, it is crucial to  
161 check that no drainage occurs during the test. A possible drainage may prohibit the use of the  
162 tested device at low ram velocity (Martin et al. 2006; Mascia et al. 2006; Perrot et al. 2007;  
163 Perrot et al. 2009; Yu et al. 1999). This can be done by measuring the densities of both the  
164 extrudates and the remaining material in the extruder.

165 Reference shear flow measurements have been independently performed under isothermal  
166 conditions (20°C) using a Brookfield Soft/Solid tester equipped with a vane geometry in an  
167 infinite cup (200x200 mm<sup>2</sup>). This type of cup is used to circumvent the wall slip artefact. The  
168 radius of the four-bladed vane geometry is 4 mm and its height is 8 mm. The shear yield  
169 stress is estimated at low shear rate using the stress growth test (Liddell and Boger 1996).  
170 Tests are conducted under a rotation rate controlled mode. The tool rotation rate is set at 0.01  
171 rad.s<sup>-1</sup>. This rotation rate is in agreement with the Lidell and Boger protocol and allows for an  
172 accurate determination of the yield stress (viscous effects neglected). As proposed by Estelle  
173 et al., the end effect is neglected (Estellé et al. 2008). The shear yield stress  $\tau_0$  is deduced using  
174 equation (1).

175 
$$\tau_0 = \frac{M}{2\pi R_v^2 h_v} \quad (1)$$

176 where  $M$  is the maximum recorded torque and  $R_v$  and  $h_v$  the vane radius and height.

177 Vane measurements provide shear yield stress of  $15 \pm 1$  kPa,  $25 \pm 2$  kPa and  $50 \pm 4$  kPa for  
178 the cement based material, the kaolin paste and the soft modelling clay, respectively. Because  
179 such materials can exhibit viscous behaviour, a Herschel-Buckley model is used in the  
180 following, making it possible to describe possible shear-thinning or shear-thickening  
181 behaviour:

182 
$$\tau = \tau_0 + k_s \dot{\gamma}^{n_s} \quad (2)$$

183 where  $\dot{\gamma}$ ,  $k_s$  and  $n_s$  are respectively the shear rate, the consistency and the flow index.

184 For the tribological behaviour, it has been shown that high yield stress fluids exhibit a friction  
185 yield stress value (Roussel and Lanos 2003). The main hypothesis is based on purely plastic  
186 flow theory where the friction stress against a steel smooth surface (roughness height,  $R_t$  up  
187 to  $3 \mu\text{m}$ ) is considered to be a fraction of the shear stress which strongly depends on the  
188 surface roughness (Avitzur 1983; Horrobin 1999). This assumption has been successfully  
189 applied to model the squeeze flow of viscoplastic materials (Sherwood and Durban 1996,  
190 1998). The friction yield stress, denoted  $K_{w0}$ , is measured using the stress growth test  
191 procedure used for the shear yield stress measurement (low rotating velocity to avoid velocity  
192 influence) replacing the vane tool by a smooth steel cylinder ( $R_C = 4$  mm and  $h_C = 4$  mm).  
193 The friction yield stress is also determined by means of the back extrusion technique. The  
194 surface roughness of the back extrusion device and the extruder barrel are equivalent (Perrot  
195 et al. 2011). The soft modelling clay exhibits a friction yield stress value of  $30 \pm 2$  kPa with  
196 both techniques. For kaolin, the measured friction yield stress value is  $9.5 \pm 0.5$  kPa with the  
197 rotating smooth cylinder and  $9 \pm 1$  kPa with the back extrusion technique. This shows that

198 both techniques provide equivalent results. The friction yield stress has not been measured for  
199 the cement-based mix.

200 Then the velocity effect is highlighted using an analogy with the rheological behaviour. A  
201 Herschel-Buckley-like equation has already been used by Benbow and Bridgwater to analyse  
202 the friction stress inside a die capillary geometry (Benbow and Bridgwater 1993). The  
203 proposed equation can be written as follows:

$$204 \quad K_w = K_{w0} + k_{fr} \cdot V_i^{n_{fr}} \quad (3)$$

205 Where  $V_i$  is the velocity at the interface,  $k_{fr}$  is the apparent friction factor and  $n_{fr}$  the friction  
206 index.

207

## 208 **2.2. High yield stress materials flowing through ram extruder**

209 In order to develop an efficient extruder to study the material flow behaviour, it is crucial to  
210 understand the flow properties of a high yield stress fluids in an axisymmetrical ram extruder.  
211 The properties of extrusion flow of high yield stress fluids have already been studied through  
212 various techniques: MRI (Götz et al. 2003; Rabideau et al. 2010; Barnes et al. 2006), positron  
213 emission particle tracking (Wildman et al. 1999), visual observations (Hill 1950; Perrot et al.  
214 2007) or numerical simulations (Horrobin 1999; Horrobin and Nedderman 1998; Jay et al.  
215 2002; Zienkiewicz et al. 1977). From those studies conducted at low shear rate, axisymmetrical  
216 extrusion flow can be divided into three parts (Fig. 1):

217 - A plug flow located in the extruder barrel where the paste seems to slip along the wall. This  
218 slippage induces friction force that should be overcome to ensure the extrusion flow.

219 - A tapered static zone where the material remains blocked around the die entry. This area is  
220 called the “dead zone”. It is assumed that the dead zone acts as a conical or tapered entry die  
221 as proposed in previous studies (Basterfield et al. 2005; Horrobin 1999).

222 - A forming zone, located inside the conical dead zone, where the material billet diameter  
223 decreases. This zone is called the “shaping zone”.

224 Thus, it appears obvious to introduce representative force for each part of the flow where  
225 material advance and forming occurs (as shown in figure 1):

226 - The shaping force required to give its final shape to the material in the shaping zone,  
227 denoted  $F_{pl}$  has been the subject of numerous studies (Avitzur 1983; Basterfield et al. 2005;  
228 Benbow and Bridgwater 1993; Gibson 1988; Hill 1950; Horrobin 1999; Horrobin and  
229 Nedderman 1998; Kobayashi and Thomsen 1963; Benbow et al. 1991) .

230 - The wall friction force  $F_{fr}$ , developed along the extruder body  $F_{fr}$  is linked to the friction  
231 stress value  $K_w$ .

232 The reference pressure is the external pressure  $P_{atm}=0$ . As described in figure 1,  $L_{dz}$  denotes  
233 the dead zone length and  $L_B$  is the friction length. The length of the dead zone is known to  
234 depend on both die and extruder body diameters. It is commonly considered that the conical  
235 dead zone is characterised by a  $\pi/4$  angle. In his study, Basterfield et al. have showed that the  
236 variation of this angle between  $\pi/3$  and  $\pi/6$  has only little effect on the extrusion pressure. As  
237 a result, the dead zone length can be modelled as follows (Basterfield et al. 2005; Mascia et  
238 al. 2006):

$$239 \quad L_{dz} = \frac{D-d}{2} \quad (4)$$

240 This assumption is in agreement with the modelling results reported in previous studies  
241 (Avitzur 1983; Basterfield et al. 2005; Benbow and Bridgwater 1993; Gibson 1988; Hill  
242 1950; Horrobin 1999; Horrobin and Nedderman 1998; Kobayashi and Thomsen 1963).  
243 However,  $L_{dz}$  is also largely influenced by the roughness of the extruder barrel and die  
244 surface. As shown by Horrobin (Horrobin 1999), the roughness of the extruder walls largely  
245 influences the dead zone length and form. As a result, it is required to measure the dead zone  
246 length after extrusion to check if eq. (4) is valid for a given extruder/material couple.

247 As shown in figure 2, the dead zone length can also be measured from the visualisation of the  
248 soft modelling clay remaining in the extruder after extrusion. The dead zone presents little  
249 curvature however we assumed that both dead zone length and form are close to the modelled  
250 conical one.

251

### 252 **2.3. Extrusion device and tests**

253 The developed ram extruder shown on figure 3 must be able to measure simultaneously both  
254 force contributions: the shaping force acting in the shaping zone and the friction forces acting  
255 on the extruder barrel.

256 The extruder barrel consists of a vertical steel barrel of 300 mm in length and 43.3 mm in  
257 diameter (denoted  $D$ ) placed under a controlled velocity load frame. The surface roughness of  
258 the barrel is  $R_t = 3 \mu\text{m}$  and represents the maximum peak to valley height of the roughness  
259 profile. A nylon ram is used to push the material inside the barrel, ensures a uniform pressure  
260 on the material and avoids liquid drainage upward the ram. This ram is equipped with a 20 kN  
261 load cell (with a 1 N accuracy) in order to measure the total extrusion force  $F$  required to  
262 overcome both frictions on the extruder barrel and shaping force in the die land.

263 The extruder barrel is placed on a novel die system which is fixed on the load frame. This  
264 system allows measuring the friction force acting along the barrel wall. The bottom of the  
265 extruder barrel and the top of the die system are both equipped with similar circular shoulders  
266 of diameter 100 mm. The shoulders are perforated by three holes (diameter 6 mm –  $120^\circ$   
267 oriented) and connected using three small perforated cylindrical 2 kN load cells which are  
268 centred on the holes (diameter 20 mm, hole diameter 6 mm, thickness 3 mm, and 1 N  
269 accuracy). Those load cells are pre-stressed by a screw-bolt system.

270 During extrusion, the material billet induces a wall friction stress on the extruder barrel which  
271 tends to move the barrel downward toward the die and consequently squeezes the three load

272 cells. The sum of the three recorded forces is considered to be equal to the friction force  $F_{fr}$   
273 acting on the extruder barrel surface.

274 Then, the shaping force can be deduced from the measured values of both the total extrusion  
275 force measured at the ram and the friction force measured by the load cells supporting the  
276 barrel.

277 The friction force measurement requires some geometrical adjustments:

278 - The length of the barrel should be greater than its diameter in order to give sufficient  
279 surface for the measurement of the friction acting on the barrel surface.

280 - The die diameter should be at least 10 times the maximum particle diameter in case of  
281 suspensions extrusion.

282 - According to eq. (4), low extrusion ratio  $d/D$  (less than 0.1) leads to high value of  $L_{dz}$ .

283 Then the dead zone may enter the friction measurement zone. In this case, use of wide  
284 bore capillary rheometer analysis should be preferred (Toplak et al. 2007).

285 This third geometrical limitation is crucial. If the dead zone is long enough to enter the barrel,  
286 the force measured by the three small load cells will not exactly correspond to the friction  
287 linked to the moving material and then will not provide an accurate value of the wall friction  
288 stress. In order to avoid this measurement artefact, a zone of 20 mm in length is placed  
289 between the exit orifice (square angle die) and the extruder barrel. With the used die  
290 diameters (denoted  $d$ ) of 8, 15 and 20 mm and using eq. (4) the computed dead zone length  
291 ranges from 11.5 to 17.65 mm (which is less than 20 mm). This ensures that the dead zone  
292 does not obstruct the movement due to the friction force at the flowing material/barrel  
293 interface.

294 The die land is 8 mm long. Wall Friction on the die land surface has to be subtracted from the  
295 measured shaping force in order to study the work done in the conical zone where the material  
296 diameter is shaped. The procedure will be presented in the shaping force analysis section.

297 A 55.3 mm diameter extrusion barrel with common square angle dies is also used in addition  
298 to the 43.3 mm diameter developed extruder. It is done to verify if the proposed data analysis  
299 depends on the extruder geometry.

300 Extrusion tests are carried out under a load frame ensuring a constant ram velocity ranging  
301 from  $0.1 \text{ mm.s}^{-1}$  to  $8.33 \text{ mm.s}^{-1}$ .

302 The material billet length is controlled and ranges from 120 to 200 mm depending on the test.  
303 Both extruder barrels can be used with three different square angle exit dies that allow for  
304 performing tests at the same extrusion ratios (i.e.  $d/D$  equals to 0.18, 0.34 and 0.45). The final  
305 volume and mass are measured and the computed apparent density is compared to the  
306 theoretical density.

307

### 308 **3. Experimental results analysis**

309

#### 310 **3.1. Extruder device validation and force data analysis**

311 An illustration of the total extrusion force versus the remaining billet length is presented in  
312 figure 4. The curve can be split into three distinct parts. The first part consists in the flow  
313 initiation. At the beginning of extrusion, the billet length is at its maximum. When the ram  
314 starts to move, the billet is compacted. As a result, the recorded extrusion force dramatically  
315 increases. We consider that this step ends when the extrusion force reaches its maximal value.  
316 Then the second part begins. The extrusion force starts to linearly decrease as the ram  
317 advances (i.e. as the remaining billet length decreases). Such particular force versus billet  
318 length evolution can be observed because the fluid is mainly plastic and remains  
319 homogeneous during the extrusion time. In addition, this linear evolution is linked to the  
320 linear decrease of the billet length due to the constant ram velocity. As the billet length  
321 linearly reduces, the surface of friction between the extruder barrel wall and the material billet

322 linearly decreases. This allows us to write the following relationship which provides us the  
323 friction stress denoted  $K_w$  from a simple force balance equation:

$$324 \quad K_w = \frac{1}{\pi \cdot D} \frac{dF}{dL_B} \quad (5)$$

325 Where  $F$  is the extrusion force and  $L_B$  is the remaining billet length as defined in figure 1.

326 The third part of the curve begins when the evolution of the force versus the billet length  
327 variation becomes non linear. This happens when the ram is near the die land area and  
328 squeezes the static zone. The recorded force can reach a constant value or can increase with  
329 the billet length decreasing. Then, the extrusion test is stopped when the billet length is equal  
330 to 5 mm.

331 The analysis of the extrusion force curve allows us to restrain the data analysis to the second  
332 part of the curve (linear decrease of the force) which provides us a friction stress value. The  
333 use of the developed extruder provides the evolution of both the friction force along the  
334 extruder barrel wall and the total extrusion force as shown in figure 5. The slopes of the  
335 friction force curve and total force curve are very close (deviation of 3%). This indicates that  
336 the two measurements provide the same information as both curves allow for the computation  
337 of the friction stress  $K_w$  using eq. (5). Testing visco-plastic non compressible material helps us  
338 to show the efficiency of the two data analysis procedures. The effects of both shaping and  
339 friction force along the extruder barrel on the total extrusion force is highlighted. Finally, with  
340 the illustration given in figure 5, the computation of the friction stress  $K_w$  with eq. 5 can be  
341 performed with the data obtained from both the total extrusion force and the measured friction  
342 force data.

343 The difference between the total extrusion force  $F$  and the friction stress  $F_{fr}$  provides a quasi-  
344 constant value which is not influenced by the ram advance as shown in figure 5. This force is  
345 considered to be equal to the shaping force  $F_{pl}$  required to reduce the billet diameter from  $D$  to  
346  $d$ . The resulting force value, denoted  $F_{pl}$  can be used to compute an elongational or a shear



347 yield stress and a velocity factor which can be linked to the consistency and the flow index of  
348 the tested material (Avitzur 1983; Basterfield et al. 2005; Benbow and Bridgwater 1993;  
349 Horrobin 1999; Kobayashi and Thomsen 1963; Mimoune and Aouadja 2004; Toplak et al.  
350 2007; Hill 1950). In addition,  $F_{pl}$  can be computed from the total extrusion force curve when  
351 the remaining billet length is equal to the dead zone length  $L_{dz}$  estimated with eq. (4). Eq. (4)  
352 provides  $L_{dz}$  value equal to 13.65 mm. At this value of billet length, the total extrusion force is  
353 equal to 716 N. This force is close to the difference between the measured total extrusion  
354 force and the measured friction force ( $F_{pl} = 693$  N – standard deviation of 3.3%).  
355 This measured value of  $F_{pl}$  is later used to study the rheological behaviour of the studied  
356 viscoplastic paste.

357

### 358 **3.2. Friction force data analysis**

359 The aim of the data analysis is to obtain the relationship between the ram velocity and the  
360 friction stress acting along the extruder barrel wall as proposed by eq. (3).

361 The friction stress value is obtained from the linear part of the evolution of the total force (or  
362 the friction force) versus the remaining billet length  $L_B$  as proposed in the previous section  
363 and as shown in figure 6 for soft modelling clay. As expected, the slope of the curve does not  
364 depend on the extrusion ratio  $d/D$  but is influenced by the ram velocity. From figure 6, it is  
365 observed that the computed slope values for different  $d/D$  and same ram velocity are very  
366 close.

367 In figure 7, computed friction stress  $K_w$  is plotted versus the ram velocity for the three tested  
368 materials. The best fit of the friction law is given for each of them. We note that the friction  
369 law given by eq. (3) is able to describe the friction behaviour of all tested materials as the  $R^2$   
370 coefficient is always higher than 0.99. In order to ensure the accuracy of the proposed method  
371 (geometry non-dependency) two extruder barrel diameters were tested. Both diameters

372 provide the same  $K_w$  values. This confirms the consistency of the proposed method as it does  
373 not depend on the extruder geometry, but seems to provide only material friction parameters.  
374 It should be noted that the extruder wall roughness is the same in both cases (same steel and  
375 same machining to obtain an average roughness of  $3 \mu\text{m}$ ). Such parameters could have large  
376 effects on the measured friction stress, but this is out of scope of the present work (Hoang et  
377 al. 2010).

378 For soft modelling clay, the obtained friction yield stress value is  $31 \pm 2 \text{ kPa}$ . This value is  
379 close to those obtained with rotating smooth cylinder and back extrusion technique given in  
380 section 2.1 (deviation of respectively 3 and 15%). For kaolin, extrusion tests provide a friction  
381 yield stress of  $8.7 \pm 1 \text{ kPa}$ . This is close the values obtained with the back extrusion technique  
382 and smooth cylinder given in section 2.1 (maximum deviation of 9%).

383

### 384 **3.3. Shaping force analysis**

385 The required pressure drop to ensure the flow of a visco-plastic fluid through an extruder die  
386 is commonly modelled by two contributions: The friction forces acting on the material/die  
387 interface and the force required for the internal bulk deformation (Benbow and Bridgwater  
388 1993; Benbow et al. 1991).

389 In our case, the recorded shaping force  $F_{pl}$  required to ensure the flow in the tapered die land  
390 (as we considered that the dead zone acts as a conical convergent die) is precisely the sum of  
391 three contributions:

- 392 - The force required for bulk deformation  $F_{pl,1}$ . It corresponds to the plastic work needed  
393 to reduce the billet diameter.
- 394 - The force required to overcome the friction on the dead zone which is considered here  
395 as a tapered die  $F_{pl,2}$ .
- 396 - The force required to overcome the friction on the exit orifice surface  $F_{pl,3}$ .

397 The analysis of the force required for the plastic work to reduce the diameter of the material  
 398 billet ( $F_{pl,i}$ ) has been the subject of numerous studies. The most used data analysis is given by  
 399 Benbow and Bridgwater (Benbow and Bridgwater 1993) and have been used successfully to  
 400 describe the flow of many materials (Alfani and Guerrini 2005; Basterfield et al. 2005; Li and  
 401 Bridgwater 2000; Mascia et al. 2006; Rabideau et al. 2010; Zhou and Li 2005a; Zhou and Li  
 402 2005b). This equation writes:

$$403 \quad F_{pl,i} = \frac{\pi D^2}{2} \cdot (\sigma_0 + \alpha V^{n_{BB}}) \ln\left(\frac{D}{d}\right) \quad (6)$$

404 Where  $\sigma_0$  is considered as the elongational yield stress,  $\alpha$  and  $n_{BB}$  are fitting parameters which  
 405 depend on the material rheological behaviour. As proposed by Adams et al. (Adams et al.  
 406 1997), for a material respecting the Von Mises criterion, the plastic elongational yield stress is  
 407 equal to  $3^{1/2}\tau_0$ . The main limitation of this model is that  $\alpha$  and  $n_{BB}$  are not material intrinsic  
 408 parameters.

409 Basterfield et al. attempt to improve the modelling of the velocity influence on the extrusion  
 410 force. This model is adapted from the Gibson equation to be able to describe viscoplastic  
 411 fluids (Basterfield et al. 2005). Assuming a spherically convergent flow (with a dead zone  
 412 angle of  $45^\circ$ ) and neglecting the contribution due to shear, the author proposed an alternative  
 413 modelling. They considered the modelling of the flow in a conical duct with an angle around  
 414  $45^\circ$  where the shear contribution can be neglected. Finally, the author derived the equation  
 415 (the transitions between conical and cylindrical flows at both the dead zone and the exit  
 416 orifice entrance are neglected):

$$417 \quad F_{pl,i} = \frac{\pi D^2}{4} \cdot \left[ 2\sqrt{3}\tau_0 \ln\left(\frac{D}{d}\right) + \frac{2k_s \sqrt{3}^{n_s+1}}{3n_s \cdot 2^{n_s}} \left(\frac{2V \cdot D^2}{d^3}\right)^{n_s} \sin \theta (1 + \cos \theta)^{n_s} (1 - (d/D)^{3n_s}) \right] \quad (7)$$

418 With  $n_s$  and  $k_s$  represent respectively the flow index and the flow consistency. We note that  
 419 when the ram velocity tends to 0, eq. (7) reduces to eq. (6). Equation (7) is finally used in the  
 420 present study to evaluate the force contribution linked with internal deformation.

421 Then, the force required to overcome the friction on the tapered dead zone surface is  
 422 computed using the modelling given by Benbow and co-authors (Benbow and Bridgwater  
 423 1993; Benbow et al. 1991). It is important to note that the friction stress acting on the dead  
 424 zone surface differ from the friction stress acting on the steel barrel (the interface roughness is  
 425 different). Moreover, in this particular case, the tribological behaviour implies a  
 426 material/material interface. As a consequence, it seems rational to consider the friction at such  
 427 interface as a thin localized shear band. Thus, as used for squeeze geometry (Sherwood and  
 428 Durban 1996, 1998; Engmann et al. 2005), it is assumed that the friction stress at the extruder  
 429 barrel is a fraction of the friction stress at the dead zone interface. We define a coefficient  $m$   
 430 as the ratio of the friction stress at the extruder barrel to the friction stress at the dead zone  
 431 interface. Moreover this coefficient  $m$  tends to  $K_{w0}/\tau_0$  when the interfacial velocity tends to 0.  
 432 The coefficient  $m$  can be expressed as follow:

$$433 \quad m = \frac{K_w(\text{steel surface})}{K_w(\text{dead zonesurface})} = \frac{K_{w0}}{\tau_0} \quad (8)$$

434 This coefficient allows us to write the friction force acting on the tapered dead zone surface.  
 435 The final expression is given by the Benbow and co-authors modelling combined with eqs.  
 436 (3) and (8) (Benbow and Bridgwater 1993; Benbow et al. 1991):

$$437 \quad F_{Pl,2} = \frac{1}{m} \frac{\pi D^2}{4} \left[ K_{w0} \cot \theta \ln \frac{d}{D} + k_{fr} V^{n_{fr}} \frac{D^{2n_{fr}}}{d^{2n_{fr}}} \left( 1 - \left( \frac{d}{D} \right)^{2n_{fr}} \right) \right] \quad (9)$$

438 It can be noted that this assumes that the slip layer behaviour is the same against the extruder  
 439 barrel and against the static zone. However, it can be considered that the power law index  
 440 taken for the material/material interface can be equal to  $n_s$  as it concerns a material/material  
 441 interface. At least, the power law index ranges between  $n_{fr}$  and  $n_s$ . This could have some  
 442 noticeable impact that would be discussed at the end of the section.

443 The third contribution which corresponds to the friction along the die orifice steel surface ( $R_t$   
444  $= 3\mu\text{m}$ ) is commonly written using a simple force balance equation:

$$445 \quad F_{p1,3} = \pi d L_O \left[ K_{w0} + k_{fr} V^{n_{fr}} \frac{D^{2n_{fr}}}{d^{2n_{fr}}} \right] \quad (10)$$

446 Finally, the shaping force  $F_{pl}$  is provided by the summation of the three contributions:

$$447 \quad F_{pl} = F_{p1,1} + F_{p1,2} + F_{p1,3} \quad (11)$$

448 For the three tested materials, it is then possible to highlight the rheological parameters (i.e.  
449  $\tau_0$ ,  $k_s$  and  $n_s$ ) which provide the best fit between experiments and modelling. Results are  
450 shown on figures 8 to 10. Those curves show that the proposed modelling is able to provide  
451 an estimation of the rheological parameters without extruder geometry dependency. This  
452 conclusion is clearly presented on figure 11 where the modelled values of the shaping force  
453  $F_{pl}$  are plotted versus the measured ones. The correlation between modelling and experiments  
454 is very good ( $R^2=0.99$ ). The Basterfield et al. analysis applied to the used die geometry and  
455 local roughness is suitable for the shaping force analysis. Rheological parameters are listed in  
456 the table 1. Shear thinning behaviour characterises the three tested materials. The comparison  
457 of the shear yield stress obtained by vane geometry (section 2.1) and Extrusion tests shows  
458 that extrusion provides close higher values for the three tested materials. The variation is  
459 more significant for of kaolin paste (35%) and cement paste (21%) and is lower for soft  
460 modelling clay (11%).

461 In order to study the effect of the power law index used to compute the friction against the  
462 dead zone, the computation is also made using  $n_s$  instead of  $n_{fr}$  in eq. (9). In this case, the  
463 obtained consistencies and flow index changes. The consistency  $k_s$  becomes 17.5 kPa.s  
464 instead of 11.5 kPa.s for the cement paste, 19.0 kPa instead of 12.8 kPa for the kaolin and 5.1  
465 instead of 16.8 kPa for the soft modelling clay. The flow index  $n_s$  remains at 0.1 for the  
466 cement paste but becomes 0.2 instead of 0.11 for the kaolin and 0.9 instead of 0.46 for the soft

467 modelling clay. Those values provide also a good correlation  $R^2=0.96$ . This shows that the  
468 assumption of the friction law against the static zone is important as it largely influences the  
469 determination of the consistency and flow index. It can be advised to use conical steel die  
470 with the same roughness as the barrel one to validate the use of  $n_{fr}$  in eq. (9).

471

## 472 **4 Conclusions**

473

474 In this study, we design a new extruder to study both the rheological and tribological  
475 properties of high yield stress materials. According to literature data, the extrusion force can  
476 be split into two contributions: the friction force acting along the extruder barrel and the  
477 shaping force in the die land. The new device is developed that makes it possible to measure  
478 both contributions. The measured friction force  $F_{fr}$  is used to study the material tribology. A  
479 simple force balance equation allows us computing the friction stress acting on the barrel  
480 wall. For the shaping force  $F_{pl}$ , the Basterfield et al. equation is adapted to our die geometry  
481 (conical dead zone which acts as a rough tapered die). The use of our experimental data bank  
482 allows us to valid our protocol of data analysis for firm materials. The method is finally used  
483 to compute the Herschel-Buckley modelling parameters of the three tested materials (soft  
484 modelling clay, kaolin clay and cement-based paste). Shear yield stress results are in  
485 agreement with those computed from vane test shear yield stress.

486

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599

600

## 601 **Table Caption**

602

603 Table 1: Rheological and tribological modelling parameters obtained with vane tests and  
604 extrusion for the three tested materials.

605

## 606 **Figure Captions**

607

608 Figure 1: Force distribution in the extrusion flow of high yield stress fluid

609 Figure 2: Dead zone length observation with soft modelling clay test extrusion (Ram velocity  
610  $1 \text{ mm.s}^{-1}$ ,  $d=16 \text{ mm}$  and  $D=43.3 \text{ mm}$ ). Comparison with modelled conical dead zones.

611 Figure 3: Friction force measurement system in the designed die.

612 Figure 4: Typical evolution of the total extrusion force versus the remaining billet length.

613 Test performed on soft modelling clay,  $D=55.3 \text{ mm}$ ;  $d/D=0.36$ ;  $V = 1 \text{ mm.s}^{-1}$ .

614 Figure 5: Evolution of the friction and total extrusion force versus the remaining billet length

615 measured with the developed extruder. Test performed on Soft modelling clay:  $D=43.3 \text{ mm}$ ,

616  $d/D=0.36$ ,  $V=1 \text{ mm.s}^{-1}$ .

617 Figure 6: Total extrusion force versus remaining billet length of soft modelling clay. Tests

618 performed for  $D = 43.3 \text{ mm}$ . Slopes of linear fitting  $\Delta F/\Delta L_B$  are: 4.12 ( $R^2=0.99$ ) for  $d = 8 \text{ mm}$

619 and  $V = 0.1 \text{ mm.s}^{-1}$ ; 5.21 ( $R^2=0.96$ ) for  $d = 8 \text{ mm}$  and  $V = 1 \text{ mm.s}^{-1}$ ; 3.85 ( $R^2=0.99$ ) for  $d = 20$

620  $\text{mm}$  and  $V = 0.1 \text{ mm.s}^{-1}$ ; 5.06 ( $R^2=0.97$ ) for  $d = 20 \text{ mm}$  and  $V = 1 \text{ mm.s}^{-1}$  ;

621 Figure 7: Friction law obtained for the three tested materials.

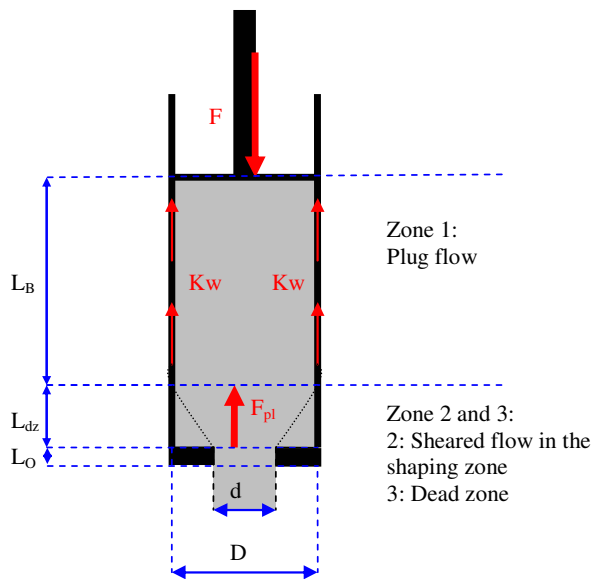
622 Figure 8: Modelled and experimental values of shaping force  $F_{pl}$  for soft modelling clay  
623 versus the ram advance.  $\tau_0 = 56.8$  kPa ;  $k_s = 16.7$  kPa.s<sup>ns</sup> ;  $n_s = 0.46$  are the values providing  
624 the best fit for the modelling. Tests performed with  $D = 44.3$  mm and  $D=55.3$  mm.

625 Figure 9: Modelled and experimental values of shaping force  $F_{pl}$  for kaolin paste versus the  
626 ram advance.  $\tau_0 = 39.1$  kPa ;  $k_s = 12.8$  kPa.s<sup>ns</sup> ;  $n_s = 0.11$  are the values providing the best fit  
627 for the modelling. Tests performed with  $D = 44.3$  mm and  $D=55.3$  mm.

628 Figure 10: Modelled and experimental values of shaping force  $F_{pl}$  for cement paste versus the  
629 ram advance.  $\tau_0 = 19.0$  kPa ;  $k_s = 11.5$  kPa.s<sup>ns</sup> ;  $n_s = 0.1$  are the values providing the best fit for  
630 the modelling. Tests performed with  $D = 44.3$  mm and  $D=55.3$  mm.

631 Figure 11: Correlation between the modelled shaping force value  $F_{pl}$  and the experimental  
632 value for the three tested materials.

633



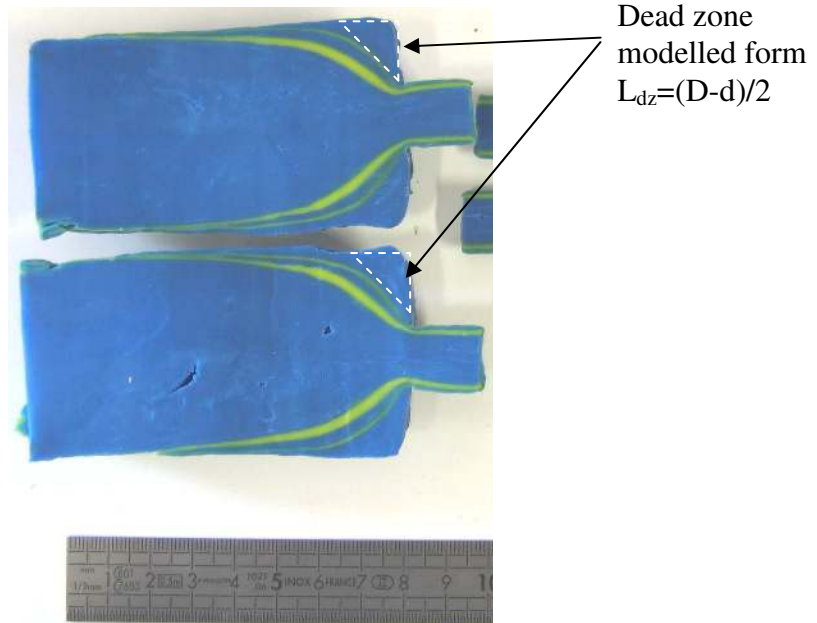
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636 Figure 1

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638



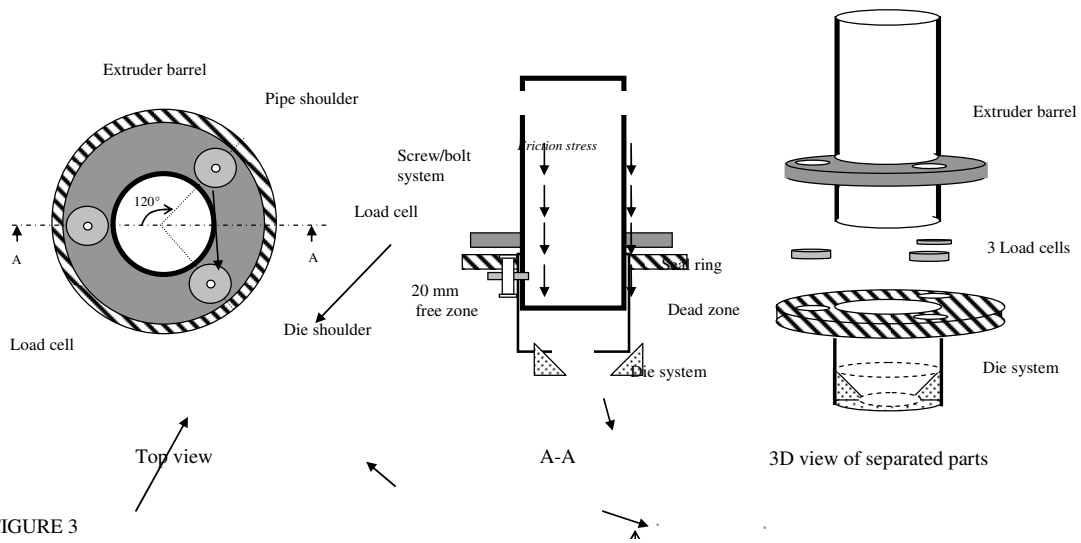
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641 FIGURE 2

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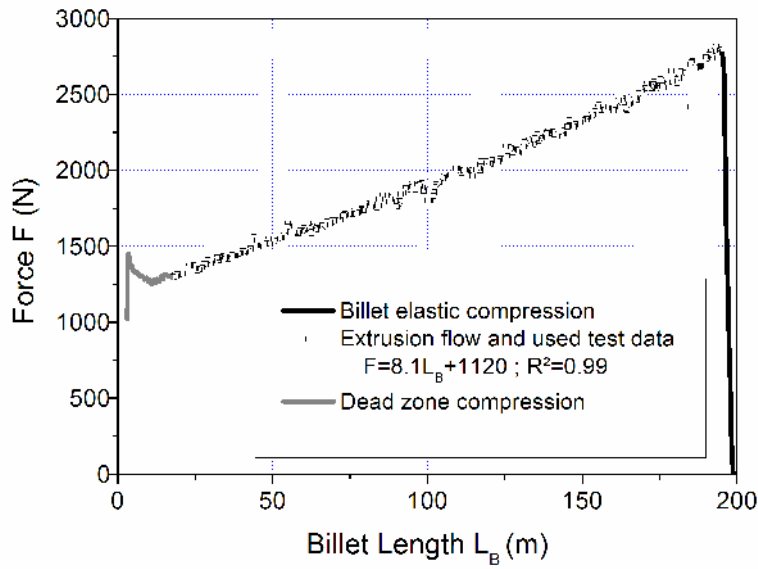
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644 FIGURE 3

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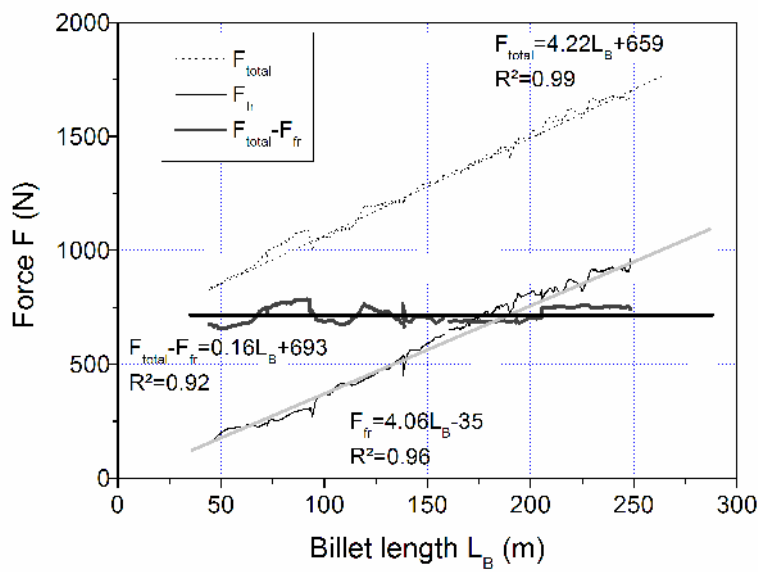
Figure 4



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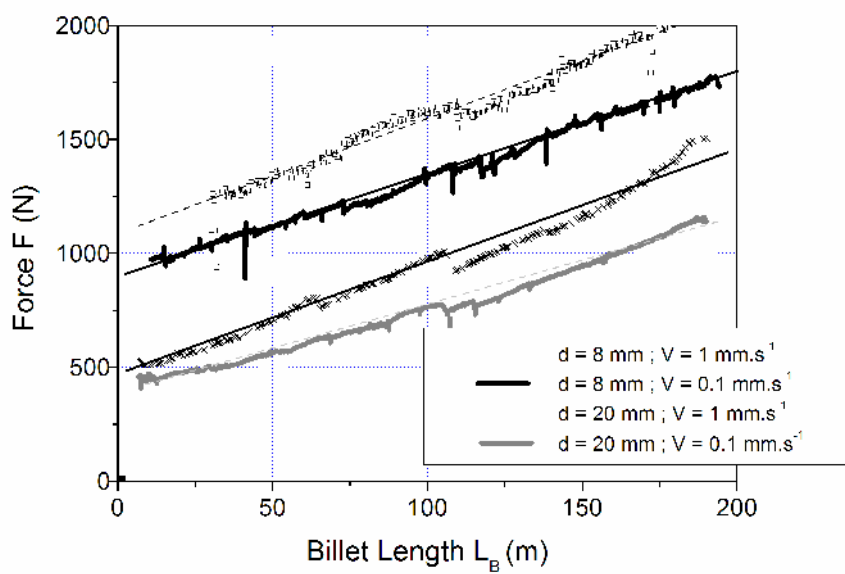
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Figure 5



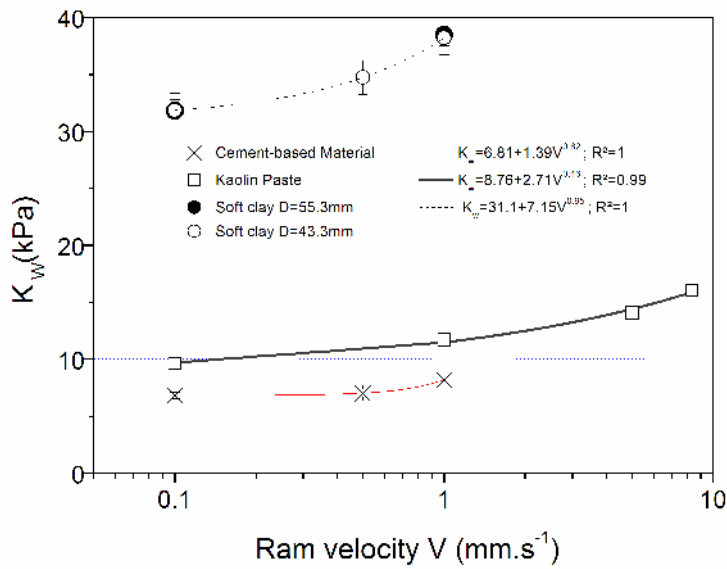
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Figure 6

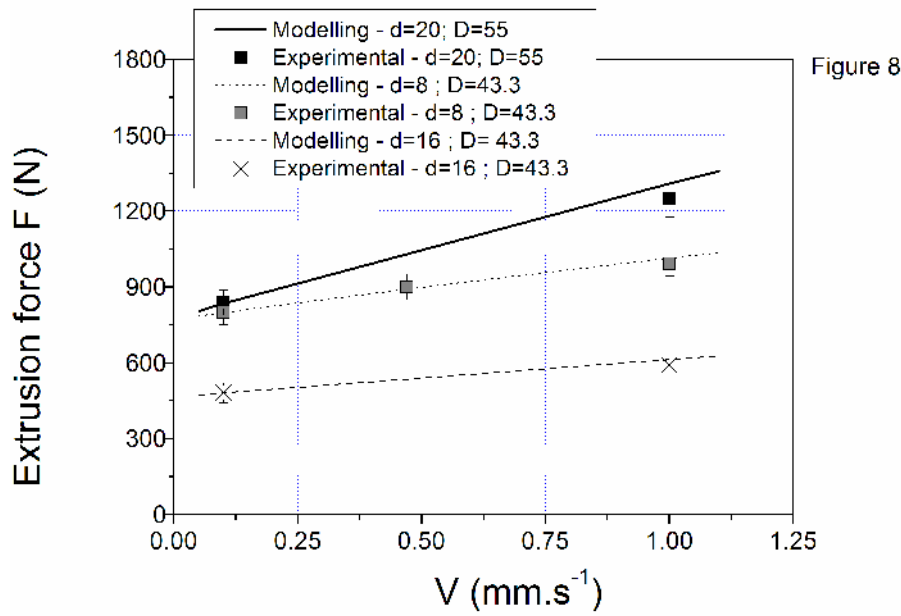


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Figure 7

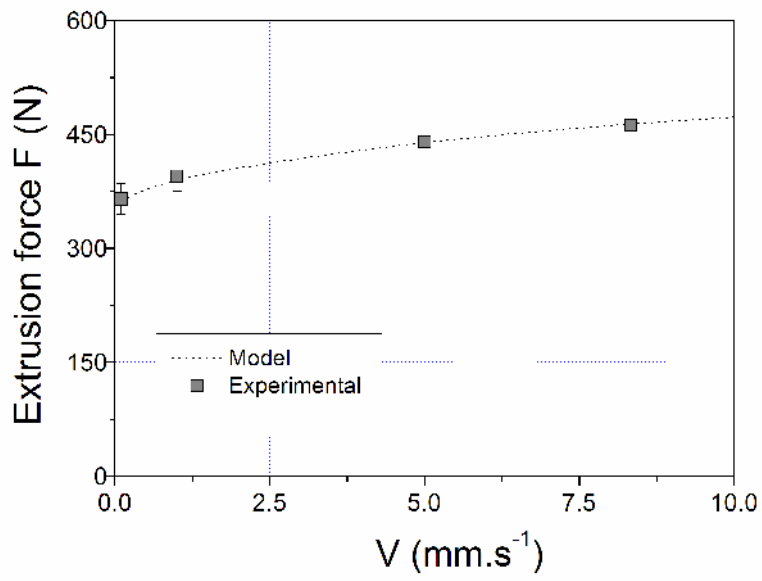


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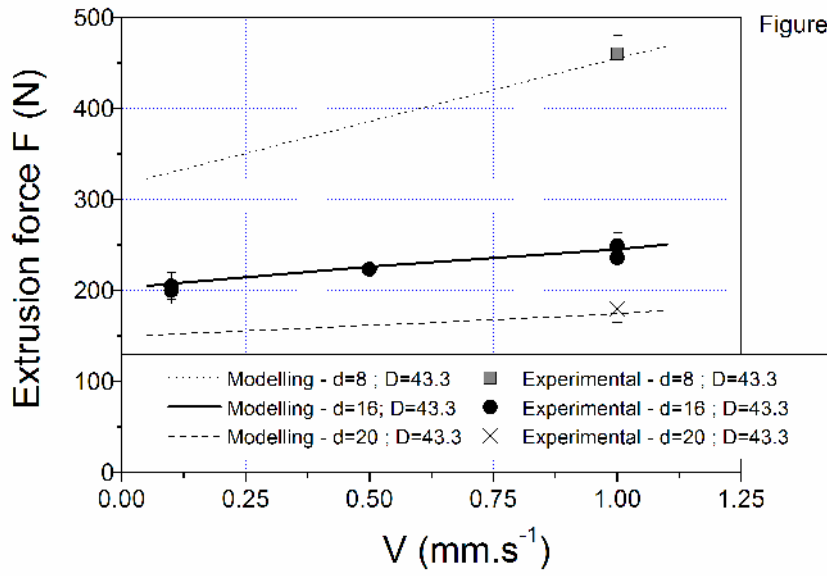
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Figure 9



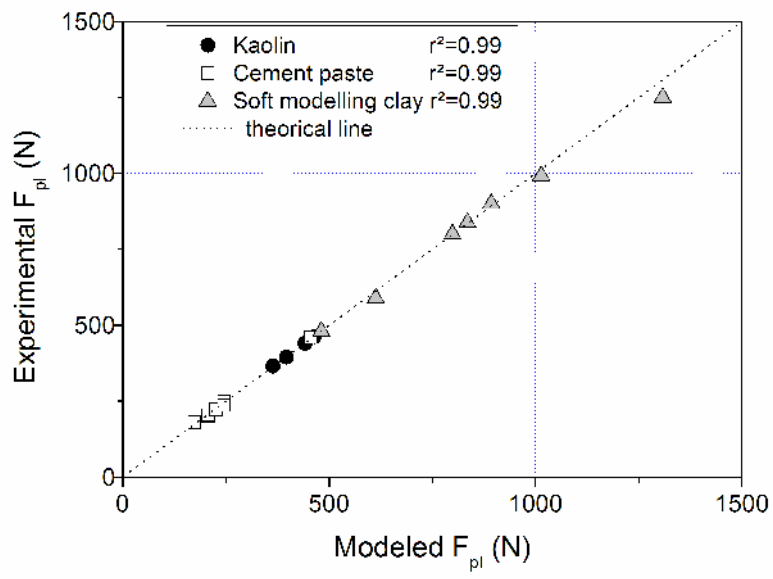
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Figure 10



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Figure 11



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