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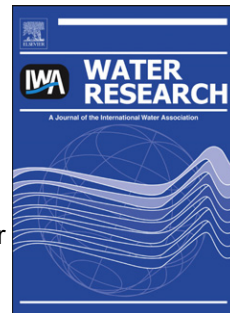
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Clear model fluids to emulate the rheological properties of thickened digested sludge

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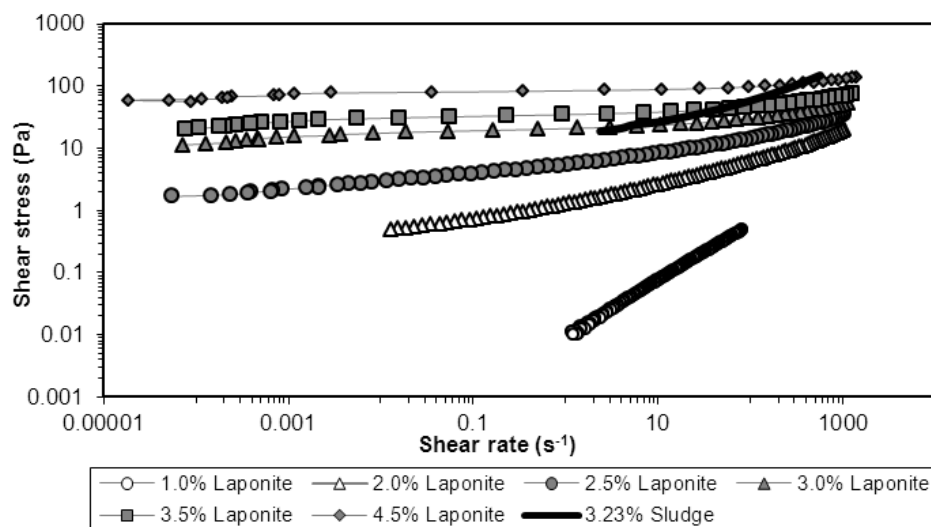
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Graphical abstract:



Anaerobic digestion is a key treatment process for solids treatment and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an appropriate transparent model fluid which can mimic the rheological behaviour of sludge is imperative for visualization of the hydrodynamic functioning of an anaerobic digester.

# CLEAR MODEL FLUIDS TO EMULATE THE RHEOLOGICAL PROPERTIES OF THICKENED DIGESTED SLUDGE

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## ABSTRACT

Optimizing flow processes in wastewater treatment plants requires that designers and operators take into account the flow properties of the sludge. Moreover, due to increasingly more stringent conditions on final disposal avenues such as landfill, composting, incineration etc., practitioners need to produce safer sludge in smaller quantities. Anaerobic digestion is a key treatment process for solids treatment and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an appropriate transparent model fluid which can mimic the rheological behaviour of sludge is imperative for visualization of the hydrodynamic functioning of an anaerobic digester.

Digested sludge is a complex material with time dependent non-Newtonian thixotropic characteristics. In steady state, it can be modelled by a basic power-law. However, for

23 short-time processes the Herschel-Bulkley model can be used to model liquid-like  
24 properties.

25 The objective of this study was to identify transparent model fluids which will mimic the  
26 behaviour of real sludge. A comparison of three model fluids, Carboxymethyl Cellose  
27 (CMC), Carbopol gel and Laponite clay revealed that these fluids could each model certain  
28 aspects of sludge behaviour. It is concluded that the rheological behaviour of sludge can be  
29 modelled using CMC in steady state flow at high shear rates, Carbopol gel for short-time  
30 flow processes and Laponite clay suspension where time dependence is dominant.

31

32 *Keywords: Thickened municipal digested sludge, rheology, thixotropic properties, Short time*  
33 *process, Steady state operation, model fluids,*

34

## 35 **1. Introduction**

36 Optimal and efficient design and operation of waste water treatment plants requires  
37 accurate prediction of the hydrodynamic functioning of the associated plant such as pumps,  
38 heat exchangers and anaerobic digester mixing. Accurate flow behaviour prediction of  
39 these engineering hydrodynamic processes requires the rheological characteristics of the  
40 sludge as input (Slatter, 2011).

41 Since the beginning of the seventies with the pioneering work of Colin (1970), and  
42 Bhattacharya (1981), rheological measurements have proved to be of great interest to  
43 quantitatively estimate the physical consistency of sewage sludge. Sewage sludge is often

44 seen as a complex mixture. The literature presents a wide variety of results: sludge is  
 45 always non-Newtonian (Campbell and Crescuollo, 1982), exhibits a yield stress (Baudez  
 46 and Coussot, 2001; Mori et al., 2006) or not (Valioulis, 1980), is shear-thinning (Chaari et  
 47 al., 2003) or thixotropic (Tixier et al., 2003). Most of the rheological models used to  
 48 describe sludge behaviour are a simple power-law (Moeller and Torres, 1997), the Bingham  
 49 model (Sozanski et al., 1997; Guibaud et al., 2003) or the Herschel-Bulkley model (Slatter,  
 50 1997; Baudez, 2001). The Sisko model, Cross viscosity model, truncated power law model  
 51 (Baudez, 2008) have been also used. Slatter (2008) has shown that the sludge rheology  
 52 plays a fundamentally important role in analysing the hydrodynamic behaviour of the  
 53 sludge, as it flows through the tertiary treatment process.

54

$$55 \quad \tau = K\dot{\gamma}^n \quad \text{Eq.1}$$

$$56 \quad \tau = \mu_B \dot{\gamma} + \tau_y \quad \text{Eq.2}$$

$$57 \quad \tau = K\dot{\gamma}^n + \tau_y \quad \text{Eq.3}$$

$$58 \quad \tau = \mu_\infty \dot{\gamma} + K\dot{\gamma}^n \quad \text{Eq.4}$$

$$59 \quad \frac{\mu - \mu_\infty}{\mu_0 - \mu_\infty} = \frac{1}{1 + K\dot{\gamma}^m} \quad \text{Eq.5}$$

$$60 \quad \frac{\tau}{\tau_c} = \left(\frac{\dot{\gamma}}{\dot{\gamma}_c}\right)^n \quad \text{Eq.6}$$

61

62 A power-law fluid (Eq.1) is a type of generalized non-Newtonian fluid, where  $\tau$  is the  
 63 shear stress,  $\dot{\gamma}$  is the shear rate, 'K' is the fluid consistency index, 'n' is the flow  
 64 behaviour index. The pseudoplastic model can be used to model the shear thinning zone of

65 the rheogram of sludge suspensions (an intermediate shear rate range). In Bingham plastic  
66 model (Eq.2), sludge is rigid when shear stress ( $\tau$ ), has a value less than a critical value  $\tau_y$ .  
67 Once the critical shear stress (or "yield stress") is exceeded, the material flows. The  
68 Herschel-Bulkley model (Eq.3) compared to the power-law model, the additional term  
69 yield stress ( $\tau_y$ ) quantifies the amount of stress that the fluid may experience before it  
70 yields and begins to flow. The physical reason for this behaviour is that sludge may contain  
71 particles or large molecules with have some kind of interaction, creating a weak solid  
72 structure and a certain amount of stress is required to break this structure and produce flow.  
73 Once the structure has been broken, the particles move with the liquid under viscous forces.  
74 If the stress is removed, the particles associate again (Chhabra and Richardson, 2008). This  
75 process also exists under shear at low shear stress (Tabuteau et al., 2006; Baudez, 2008).  
76 The advantage associated with employing the Herschel-Bulkley fluid model (Eq.3)  
77 describes both pseudoplastic (rheogram curvature) as well as the Bingham model (yield  
78 stress) (Slatter, 1999); however, the model does not take into account the Newtonian  
79 asymptotes at low and high shear rates (Slatter, 1999). At high shear rate, the model  
80 suggested by Baudez et al. (2011a), can be seen as a start to improve rheological  
81 characterisation of sludge. Both Bingham plastic and Herschel-Bulkley models can  
82 characterize the sludge behaviour from rest to flow. It is also important to note that in some  
83 cases, sludge may present slight thixotropic behaviour (Tabuteau et al., 2006, Terashima et  
84 al, 2009), which causes hysteresis on flow curves although (Baudez, 2006) demonstrated  
85 that hysteresis loop inside the rheogram cannot be used to define the thixotropic character

86 of a sludge. This is most frequently explained by the time-dependent disintegration of  
 87 internal structure of a liquid suspension as a result of the application of shear stress.

88 The Sisko model (Eq.4) can be used to characterize the flow behaviour of sludge in the  
 89 intermediate and high shear rates range, where the apparent viscosity tends to a limiting  
 90 value (plateau) (Mori et al. , 2006). The Cross viscosity fluid model (Eq.5) is a four  
 91 parameter model that relates the fitting parameters  $K$  (parameter with dimension of time)  
 92 and  $m$  (dimensionless material parameter) to the zero shear viscosity,  $\mu_0$  and infinite shear  
 93 viscosity,  $\mu_\infty$  (Sybilski, 2011). Eq.5 is further simplified and presented in Eq. 6 because  
 94 high shear rates cannot be accurately measured and thus the value of  $\mu_\infty$  cannot be  
 95 determined; it can also be assumed that  $\mu > \mu_\infty$  (Sybilski, 2011).

$$96 \quad \mu = \frac{\mu_0}{1 + K\dot{\gamma}^m} \quad \text{Eq.6}$$

97

98 Baudez (2008) defined a critical shear stress,  $\dot{\tau}_c$  (with corresponding critical shear rate,  $\dot{\gamma}_c$ )  
 99 which below that the solid structure rebuilds even under shear. In fact, below the critical  
 100 shear stress, colloidal forces tend to rebuild the solid structure (physical aging) and shearing  
 101 forces tend to break the solid structure (shear rejuvenation). As soon as the critical shear is  
 102 reached, the solid structure is completely collapsed, and fluid starts flowing which the  
 103 relationship between the shear rate and the shear stress can be defined with a truncated  
 104 power-law (Eq.6) model. He also correlates the higher degree of thixotropy of the material  
 105 to stronger interactions between the solid particles.

106



107 Sludge rheology is complex and always evolves with time due to ageing and microbial  
108 activity. The time-dependent characteristics of sludge rheology make the measurement of  
109 physical parameters difficult and the results obtained are often unreliable because of this.  
110 Therefore it cannot be used as a reference material for industrial process design or  
111 controlled experiments. As a result of this, researchers have been trying to find a suitable  
112 transparent model fluid that mimics the behaviour of sludge. Most studies have focussed on  
113 the characterisation and modelling of activated sludge, but there are very few studies on  
114 digested sludge. For activated sludge, the proxy materials that have been studied so far  
115 include kaolin suspension for the yield stress determination (Spinosa and Lotito, 2003)  
116 polymeric gels (Legrand et al., 1998), polyvinyl chloride (PVC) suspensions (Bongiovanni,  
117 1998) and polystyrene latex (Sanin and Vesilind, 1996; Örmeci and Vesilind, 2000).

118  
119 To model the flow properties of activated sludge in liquid regime, kaolin suspensions are  
120 often used (Héritier et al., 2010) due to its similarity in rheological behaviour and can be  
121 described by a Herschel-Bulkley model (Baudez, 2001; Masalova et al., 2006). Sanin and  
122 Vesilind (1996) reported a synthetic sludge made up of polystyrene latex particles and  
123 polysaccharide-alginate. Their experiments demonstrated that it is possible to create  
124 chemical surrogate sludge by using bacteria like particles, extracellular resemble  
125 polysaccharides, and cations common to activated sludge at typical quantities. However,  
126 their synthetic sludge shows poor flocculation behaviour due to the absence of filamentous  
127 microorganism which is the backbone for overall floc structure in activated sludge (Nguyen  
128 et al., 2007a). Örmeci and Vesilind (2000) improved the characteristic of synthetic sludge  
129 by adding cellulose fibres to simulate the filamentous microorganisms in activated sludge,

130 which results in stronger floc formation and better dewaterability as well as settling, though  
131 the fibres are hard to dissolve in water (Baudez et al., 2007). They observed that both  
132 calcium ions and alginate are important to promote the floc formation in synthetic sludge,  
133 which is later confirmed by Nguyen et al. (2007a; 2007b; 2008,). Dursun et al. (2004)  
134 compare the physical characteristic of the synthetic sludge to activated sludge. They noted  
135 that the synthetic sludge may be an adequate surrogate in terms of electrokinetic and  
136 rheological properties but does not duplicate the properties and conditionability of activated  
137 sludge. Therefore the application of this synthetic sludge is only valid for qualitative or  
138 mechanics studies. Baudez et al. (2007) suggested that it is necessary to consider granular  
139 arrangement by adding micro-aggregates (Jorand et al., 1995) into synthetic sludge as real  
140 sludge does not contain individual bacterial cells only.

141  
142 Baudez et al. (2007) commented that standard methodologies should be developed to  
143 prepare synthetic suspensions that mimic behaviour of both inorganic and organic sludge  
144 for lab testing. They showed that a mixture of kaolin and calcite/quartz sand in water with  
145 relative ratio ranges from 90/10% to 75/25% was able to well describe the behaviour of real  
146 inorganic sludge. As for organic sludge, it is developed based on the recipe by Müller and  
147 Dentel (2002) and Dursun et al. (2004) and consists of alginate, cellulose, yeast, bovine,  
148 serum albumin, stearic acid, and calcium as well as potassium ions in water. However,  
149 preparation of such recipe could be difficult as it requires one to have extensive knowledge  
150 on surface chemistry and colloidal system to ensure the synthetic sludge behaves exactly  
151 the same as the real sludge. Also, rheological measurement needs to be performed on the  
152 synthetic sludge to examine its behaviour to compare with activated sludge.

153

154 It is known that anaerobic digestion is a key treatment process for solids treatment, and  
155 odour and pathogen reduction. Due to the inherent opacity of sludge, it is impossible to  
156 visualize the mixing and flow patterns inside an anaerobic digester. Therefore, choosing an  
157 appropriate transparent model fluid that can mimic the rheological behaviour of sludge is  
158 imperative for visualization of the hydrodynamic functioning of an anaerobic digester. The  
159 idea of model fluid for sludge is to find a material that is less complicated but at the same  
160 time still be able to simulate the sludge behaviour. There is no study to find the  
161 representative, safe and non-smelly fluid which could mimic the rheological behaviour of  
162 sludge. Rheological properties of Carbopol, CMC and Laponite have been investigated in  
163 the past decades for various applications in food science and surface coating but very few  
164 compared their properties to the behaviour of sludge. Baudez et al. (2011b) recently have  
165 examined that the similarity between soft glassy material and sludge, based on its dynamic  
166 visco-elastic behaviour, i.e. the storage modulus ( $G'$ ) and loss modulus ( $G''$ ), in the  
167 temperature range of 10 to 60°C using strain and stress sweep tests. This work provides the  
168 basis for future work in sludge characterisation utilizing the known characteristic of soft  
169 glassy materials. Bonn (1999; 2002) has shown that Laponite is soft glassy material. This  
170 might be because Laponite is also a complex fluid like sludge, which complicates the  
171 rheological analysis. The experimental results obtained here do indicate some similarities  
172 between the rheological behaviour of Laponite and sludge under the influence of solid  
173 suspension. But due to thixotropic behaviour of Laponite, in order to study the yield stress  
174 effect of sludge, for example, on mixing system, a simple system is required. Coussot et al.

175 (2009) also highlighted that the Carbopol gel is a type of material which can be an excellent  
176 model of yield stress fluid. Therefore, Carbopol has been chosen to study for yield stress  
177 effect of sludge. CMC sample also is a good example of fluid with no yield stress which  
178 can be used to study the sludge property once a critical stress was applied and the yield  
179 stress of sludge overcome. This paper aims to identify model fluids which will mimic the  
180 behaviour of real sludge. These materials are widely available, non toxic and less  
181 complicated to handle when use for sample preparations. Besides that, the rheological  
182 properties of these materials have been well documented and therefore provide a strong  
183 database to be adapted for sludge characterization. This enable a standard methods for  
184 sludge characterization, as proposed by European Committee for Standardization (CEN)  
185 (Spinosa, 2001), to be established so that technical language, methods and practice used  
186 among researchers across the world for sludge characterization can be synchronized. For  
187 this purpose the flow property and thixotropic behaviour of real sludge were measured then  
188 by measuring similar property in model fluids, the model fluid could represent sludge  
189 rheological behaviour in high shear, low shear. The best fit rheological model for  
190 experimental data of model fluids and sludge is also presented.

## 191 **2. MATERIAL AND METHODS**

### 192 **2.1 Material:**

193 The digested sludge obtained from Mount Martha waste water treatment plant with total  
194 solid suspended of 1.65% was concentrated to 3.23% TSS via vacuum filtration (Buchner  
195 Funnel) for 6 hours. The model fluids which were used in this study were CMC, Carbopol,  
196 and Laponite. Carboxymethylcellulose sodium salt (CMC) purchased from Sigma Aldrich

197 ( $M_w$  700 000, D.S 0.9). Various aqueous CMC solutions were prepared by dissolving the  
198 correct amount of CMC in deionised water at room temperature with continuous stirring  
199 using an impeller mixer (Eurostar, power control Visc (Mixer)) at 1000 RPM. Carbopol  
200 @940 purchased from ACROS ORGANICS. Laponite RD purchased from Southern Clay  
201 Products. Various concentrations of Carbopol were prepared by dissolving the correct  
202 amount of Carbopol in deionised water at room temperature with continuous stirring using  
203 an impeller mixer (Eurostar, power control Visc (Mixer)) at 1000 RPM. In order to  
204 neutralize these Carbopol solutions, 1M NaOH was added in a drop like manner whilst the  
205 pH was simultaneously measured until a pH of 7 was reached. 1.7, 9.2, 19.0, 28.6 mL  
206 NaOH are required neutralizing 200g of 0.1, 0.5, 1, and 1.5% Carbopol, respectively. The  
207 concentration of Laponite was prepared by dissolving the specific amount of Laponite  
208 powder into the corresponding amount of tap water whilst continuous mixing it  
209 simultaneously. After 48hr, the Laponite suspension made a gel structure.

## 210 **2.2 Method:**

211 The flow curve for thickened digested sludge at  $25(\pm 1.0)^\circ\text{C}$  was determined using the  
212 Dynamic Stress Rheometer (SR200, Rheometrics) fitted with a couette geometry (inner  
213 diameter: 29mm, outer diameter: 32mm, length: 44mm). A constant shear stress was first  
214 imposed in order to pre-shear the sludge for 15 minutes and a relaxation time of 1 minute  
215 was allowed. The flow curve was then obtained by imposing an increasing stress that  
216 coincided with a shear rate less than  $1000\text{s}^{-1}$ . This measurement was repeated for 3 different  
217 samples coming from the same withdrawal at the WWTP in order to determine the  
218 consistency of the results. The flow curve for model fluids at a constant temperature

219 (25±1.0 °C) were carried out by applying a shear stress within the range of 0.017 to 20 Pa  
220 for CMC and 30 to 300 Pa for Carbopol using the SR 200 rheometer. For higher shear rate,  
221 shear rate controlled rheometer (Rheologica instruments AB, Scientex) with the couette  
222 geometry (gap diameter = 3.0 mm) for 50 to 500 RPM range was used as the SR200 was  
223 reached to its upper limit for torque measurement in the high shear rate range.

224 The thixotropic property of the concentrated digested sludge was measured at 25°C by  
225 applying a pre-shear (corresponding to 200s<sup>-1</sup>) for a duration of 15 minutes. Then without  
226 relaxation time, a first shear stress corresponding to a shear rate of 100s<sup>-1</sup> was applied for  
227 15 minutes; a second shear stress corresponding to 5s<sup>-1</sup> was then applied and the first shear  
228 stress was reapplied, each for duration of 15 minutes. The resulting shear rate versus time  
229 graph was analyzed in order to describe the thixotropy of digested sludge. The thixotropic  
230 property of Laponite at a constant temperature of 25 ±1.0 °C, was measured the same way  
231 of sludge measurement by applying a pre-shear (corresponding to 400s<sup>-1</sup>) for 15 minutes  
232 then a first shear stress corresponding to a shear rate of 350s<sup>-1</sup> for 15 minutes; a second  
233 shear stress corresponding to 100s<sup>-1</sup> for 15 minutes and reapplying the first shear again for  
234 another 15 minutes. The resulting shear rate versus time graph was analyzed (Amemiya and  
235 Shoemaker, 1992; Baudez, 2008).

### 236 **3. RESULT AND DISCUSSION**

237 The viscosity flow curve for thickened digested sludge at 3.23% is presented in Figure 1.  
238 As illustrated in Figure 1, this sludge sample can be modelled with Herschel-Bulkley model  
239 with shear thinning behaviour which is consistent with other researcher's observation

240 (Chaari et al., 2003). The parameters of model are  $\tau_y = 1.504 Pa$  ,  $K=0.296 Pa.s^n$ , and  
241  $n=0.606$ . Figure 2 shows the thixotropic characteristic of thickened digested sludge at  
242  $25^\circ C$ .

243 Fig.1: Viscosity curve of thickened digested sludge with 3.23%TSS at  $25^\circ C$

244 Fig.2: Thixotropic behaviour of thickened digested sludge with 3.23%TSS at  $25^\circ C$

245

### 246 **3.1 Similarity of sludge rheological behaviour during steady state operation** 247 **to the rheology of CMC solution**

248 If only high shear rate range (above  $20s^{-1}$ ) of the sludge viscosity curve (Fig.1) is to be  
249 considered, one straight line or in another word the power law model is adequate for  
250 modelling experimental data. Therefore, sludge at high shear rates and at steady state  
251 operation can be modelled similar to CMC at shear rate higher than  $10 s^{-1}$  with a power-law  
252 model although Cross model decies whole shear rate range of CMC solution. Figures 3  
253 show the viscosity curves of different CMC concentrations, respectively.

254 Fig.3: Viscosity Curves of different CMC concentrations

255 Figure 3 was obtained by combining both SR200 and Rheologica data. But before merging  
256 these data, it is required that the raw data from the rheometer to be corrected for the effect  
257 of non-Newtonian flow behaviour in the wide gap measurement technique. The true shear  
258 rate was determined through the technique described by Coussot (2005). After finding true  
259 shear rate and shear stress of fluid in the wide gap of couette geometry, the data was  
260 combined with the raw data of SR200 and then the Cross viscosity model (Sybilski, 2011)

261 was used to describe the flow curve of CMC. In order to determine the Cross model  
 262 parameters, the error between the predicted viscosity (using the Cross Model) and the  
 263 measured viscosity according to Eq.7, was minimised. Table 1 shows the Cross model  
 264 viscosity parameter for various CMC concentrations.

$$265 \quad SSE = \frac{(\mu_{\text{Predicted}} - \mu_{\text{Measured}})^2}{(\mu_{\text{Measured}})^2} \quad \text{Eq.7}$$

266 Tab.1: Summary of Cross Model viscosity parameters

CMC (Wt.%)	m	K	$\mu_0$
0.5	0.633	0.024	0.373
1	0.603	0.273	3.322
1.5	0.309	2.313	36.250

267  
 268 Figure 3 demonstrates that CMC exhibits shear thinning behaviour similar to sludge at high  
 269 shear rate and follows the Cross model for power law fluids (Barnes, 1999). The error  
 270 associated with measured data and Cross model calculations is relatively low suggesting  
 271 that the Cross model can be used to model the fluid properties of aqueous CMC solutions at  
 272 low concentrations (Benchabane and Bekkour, 2008). The plateau illustrated in Figure 3  
 273 suggests that the fluid behaves as a Newtonian fluid at low shear (Barnes and Walters,  
 274 1985), and also behaves as a non-Newtonian fluid following the power law model at higher  
 275 shear rates.

276



277 **3.2 Similarity of sludge rheological behaviour during short time process**  
278 **operation to rheology of Carbopol fluid**

279 The issue of whether yield stress really exists is still debatable until today. The main reason  
280 is that no equipment, so far, allows researchers to measure the shear stress of sludge at very  
281 low shear rate without being affected by the wall-slip or end effects. Besides that, the  
282 concept of yield stress is not well-defined. There is variation in terms of rheological model  
283 and experimental method used among researchers to determine yield stress points of a  
284 material. It is generally accepted that a rheological model that includes a yield stress term  
285 can be used to represent the flow behavior of sludge over a limited shear rate range, but  
286 does not necessary indicate that the sludge is a yield stress fluid (Barnes, 1999). Based on a  
287 review paper by Seyssiecq et al. (2003), with apparatus being more advanced, it is  
288 commonly admitted among researchers that yield stress does exist in aggregated  
289 concentrated sludge.

290 Yield stress is defined as minimum stress that needs to be applied for a material to flow  
291 steadily. It is generally agreed among researchers that yield stress tends to increase as the  
292 solid concentration of sludge becomes higher, even for pre-treated or conditioned sludge  
293 (Mikkelsen, 2001; Forster, 2002; Seyssiecq et al., 2003; Spinosa and Lotito, 2003; Wilen et  
294 al., 2003, Khongnakorn et al., 2010;)

295 The viscosity curve for Carbopol in Figure 4 is plotted by using data obtained from SR200.  
296 The Herschel-Bulkley model was fitted to the data and its parameters are presented in Table  
297 2. It is evident that Carbopol at the studied concentrations follows the Herschel-Bulkley  
298 Fluid model with shear thinning behaviour as illustrated a value below one for 'n'. A

299 critical yield stress must be applied in order for Carbopol to flow, which is evident for all  
 300 Carbopol concentrations; this also increases with increasing concentration. Coussot et al.  
 301 (2009) also highlighted Carbopol gel as a type of material which can be an excellent model  
 302 for yield stress fluid. Therefore Carbopol will be a suitable material for studying the yield  
 303 stress effect on sludge for different short time process such as pumping.

304 Tab.2: Rheological properties of Carbopol using the Herschel-Bulkley fluid Model

Carbopol (Wt. %)	n	K	$\tau_y$
0.1	0.513	3.2	24.50
0.5	0.502	27.50	118.33
1.0	0.484	37.20	143.20
1.5	0.456	53.67	169.33

305

306 Fig.4: Viscosity curves for 0.1% ( $\diamond$ ), 0.5% ( $\Delta$ ), 1.0% ( $\square$ ) and 1.5% ( $\circ$ ) Carbopol samples

307 This study's observation is consistent with the results of the other researchers (Curran et al.,  
 308 2002; Putz and Burghelca, 2009; Gomez et al., 2010,).

309

310 These results are compared with other researchers who also have investigated the  
 311 rheological properties of Carbopol in their works in various applications (Table 3). It is  
 312 agreed among researchers that the Herschel-Bulkley model fluid is a suitable model to  
 313 describe the behaviour of Carbopol solution at various concentrations (Roberts and Barnes,

314 2001; Kim et al., 2003; Coussot et al. 2009; Gomez et al., 2010), but none of them use  
 315 Carbopol as model fluid for sludge.

316

317 Tab.3: Rheological characterization of Carbopol for various applications

Author	Year	Application	Suspension concentration (%)
Robert and Barnes	2001	Examine slip effect on Carbopol dispersion	0.045 – 1.00
Kim et al.	2003	Evaluate viscoelastic property of Carbopol	0.1 – 4.0
Gomez et al.	2010	Model fluid for pulp mixing	0.1 – 1.5

318

319 In general, behaviour of Carbopol may seem similar to sludge as it is shear thinning and  
 320 exhibit yield stress. As suspension concentration increases, both viscosity as well as yield  
 321 stress of sludge and Carbopol increases. However, it is well known that Carbopol is a more  
 322 stable system, i.e. non-thixotropy (Moller et al., 2009) when compare to sludge, therefore  
 323 making it suitable to be used as a model fluid for sludge when thixotropic property is not a  
 324 major concern.

### 325 **3.2 Time characteristic of sludge**

326 Figure 2 demonstrates that the property of sludge depends on shear history and processes  
 327 that the sludge undergoes. Indeed, thixotropy demonstrates time-dependency of sludge  
 328 structural changes at a given shear rate which is indication of a competition between  
 329 flocculation and de-flocculation (Baudez, 2008). In practice, there is a possibility of

330 clogging the sludge transportation pipeline if the wall shear stress is to be below critical  
331 shear stress (yield stress). Because at shear stress below yield stress, the earlier  
332 deflocculated sludge will re-flocculates , and solid characteristic becomes predominant  
333 during shear (Baudez, 2008) and gradually solid deposition builds up from wall to the  
334 centre of the pipe. In Figure 2, there is 17% difference between shear rate data obtained for  
335 the same imposed shear stress (at the first and final stage) corresponding to  $100 \text{ s}^{-1}$  shear  
336 rate.. This indicates that the sludge flocculated again when the shear rate dropped from  $100$   
337  $\text{ s}^{-1}$  to  $5 \text{ s}^{-1}$  and for this reason, the response for final and the same shear stress is not the  
338 same as of the first one. Because before the first shear at  $100 \text{ s}^{-1}$ , sludge undergoes a pre-  
339 shearing stress at  $200 \text{ s}^{-1}$ , but before the final shear at  $100 \text{ s}^{-1}$ , sludge undergoes shear at  $5 \text{ s}^{-1}$ ,  
340 which demonstrates the thixotropic behaviour of the sludge. This characteristic of sludge  
341 is important when sludge undergoes different shearing stress. For example, there is a  
342 difference between the shearing stress in a heat exchanger and inside of anaerobic digester.  
343 Therefore, it is important to consider thixotropic property (flocculation and de-flocculation)  
344 of sludge for all engineering design such as pipeline design for pumping or heating purpose  
345 in order to prevent clogging; mixing system design in order to prevent deposition of sludge  
346 at the bottom of tank. Using external heat exchangers is common in wastewater treatment  
347 plant in order to keep anaerobic digester temperature constant at  $37^\circ\text{C}$  or  $55^\circ\text{C}$  depending  
348 on mesophilic or thermophilic condition. Thus, choosing a clear model fluid which can  
349 capture the time dependent characteristics of the sludge in the anaerobic digester is  
350 important.

351 The viscosity curve of Laponite clay can also be modelled with the Herschel-Bulkley  
 352 model which is presented in Figure 5. Like Carbopol, Laponite presents shear thinning  
 353 behaviour ( $n < 1$ ) with a critical shear stress and rate which below that there is no flow. In  
 354 Figure 5, it is evident that Laponite viscosity increases as the suspension concentration is  
 355 increased. Table 5 demonstrates that the parameters of this model ( 'K', 'n' and  $\tau_y$  ).

356

357

358

359 Fig. 5: Viscosity curve for different concentration of Laponite: 1.0% (●), 2.0% (X), 2.5%  
 360 (○), 3.0% (Δ), 3.5% (◇) and 4.5 % (□)

361

362 Tab.5. Rheological properties of Laponite using the Herschel-Bulkley fluid Model

Laponite (Wt. %)	n	K	$\tau_y$
1.0	0.90	0.01	0.001
2.0	0.38	0.78	0.587
2.5	0.36	2.06	3.205
3.0	0.34	2.41	17.614
3.5	0.32	3.96	29.818
4.5	0.32	5.2	77.667

363

364 Figure 6 clearly shows that the Laponite possesses thixotropic properties. In this paper, it is

365 observed that the thixotropic effect cannot be correlated to the rest time or even suspension

366 concentration. There is no clear trend or pattern in suggesting whether thixotropy becomes  
367 more apparent or not as the rest-time or suspension concentration is increased. Also it is  
368 noticed that the thixotropic test results are not reproducible and may vary by several orders  
369 of magnitude even if the experimental conditions were to remain the same. The thixotropic  
370 effect of Laponite might not be apparent over a short rest time difference. Many researchers  
371 have investigated the thixotropic properties of Laponite on daily basis or over the course of  
372 a longer period (Joshi et al., 2008; Labanda and Llorens, 2008) based on the change in  
373 rheological properties (Knabel et al., 2000; Labanda and Llorens, 2008).

374

375 Fig.6: Thixotropic behaviour of Laponite with 30 minutes rest time between each shear:

376 1.0% (X), 2.0% ( $\Delta$ ), 2.5% ( $\circ$ ), 3.0( $\diamond$ ) and 3.5 % ( $\square$ ) and 4.5 % (--)

377

378 It seems that Laponite has proved to be a better model fluid for sludge compared to  
379 Carbopol as it can capture both the yield stress and thixotropic properties of digested  
380 sludge, as well as presenting with similar rheogram curvature, as is demonstrated in Figure  
381 7. Unlike Carbopol, Laponite may be a temperature sensitive fluid, as indicated by the  
382 work of Shukla and Joshi (2008), which is important as sludge rheology is affected by  
383 thermal history (Baudez et al., 2011b). Nevertheless, further investigations need to be done  
384 to examine the effect of physical chemistry such as surface charge or temperature on the  
385 rheology of Laponite and compare it to sludge.

386

387 Fig.7: Comparative flow curves of all model fluids a) Carbopol, b) Laponite and c) CMC)

388

with 3.23% digested sludge

389

**390 4. CONCLUSION**

391 A thickened digested sludge has been rheologically characterized and the results showed  
392 shear-thinning with time dependent (thixotropic characteristics) behaviour for samples  
393 taken from one of Melbourne's wastewater treatment plants. Candidate transparent fluid  
394 model materials to mimic the sludge behaviour were also rheologically characterised. In  
395 comparison between real sludge and model fluid, it was concluded that the real sludge in  
396 steady state and at high shear rate operation such as pipe flow, mixing, aeration can be  
397 modelled by a basic power-law approach, similar to CMC. Also, Carbopol can be a good  
398 proxy for real sludge for short-time processes such as pumping and its liquid-like property  
399 can be modelled with the Herschel-Bulkley model. The time characteristic of sludge can be  
400 modelled using Laponite clay, but further study is required at different temperatures in  
401 order ensure that Laponite can emulate behavioural changes due to temperature variation.

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626

**Research highlights:**

Real sludge can be modelled:

- As non-Newtonian shear thinning thixotropic material
- In steady state, and at high shear rate operation with a power-law similar to CMC
- In short-time processes such as pumping with carbopol and the Herschel-Bulkley model
- With laponite for thixotropic property characterization

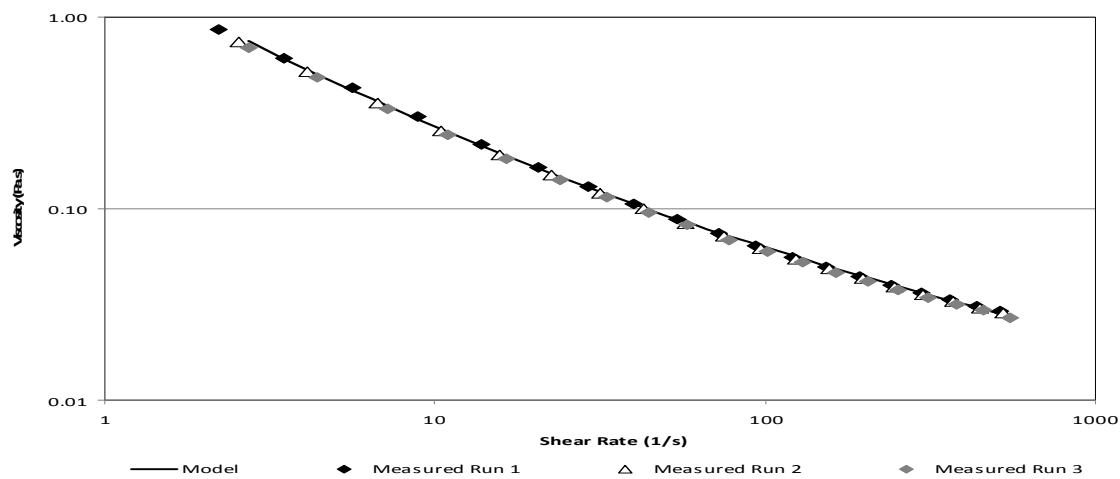


Fig.1: Viscosity curve of thickened digested sludge with 3.23% TSS at 25°C

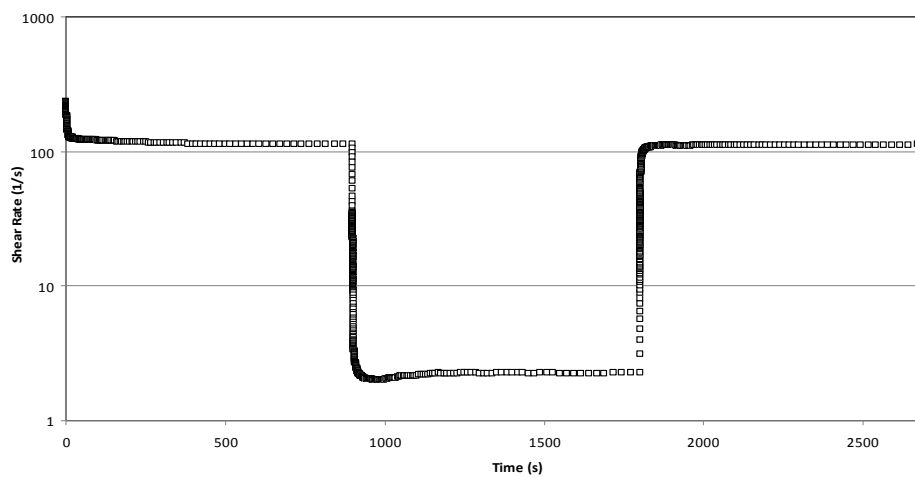


Fig.2: Thixotropic behaviour of thickened digested sludge with 3.23% TSS at 25°C

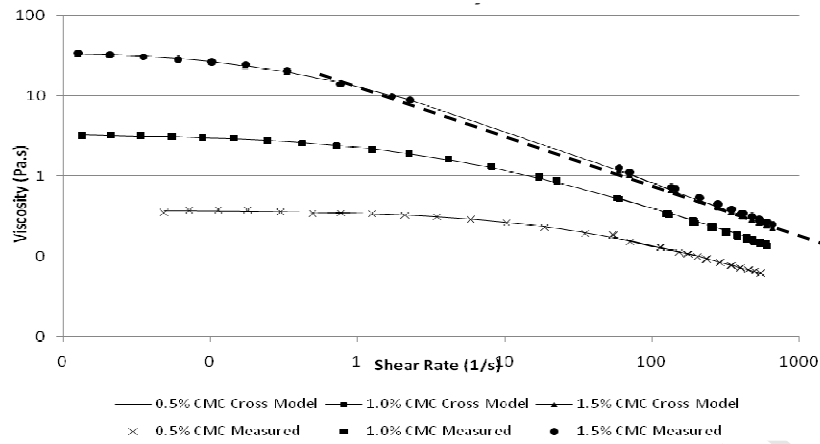


Fig.3: Viscosity Curve of different CMC concentrations

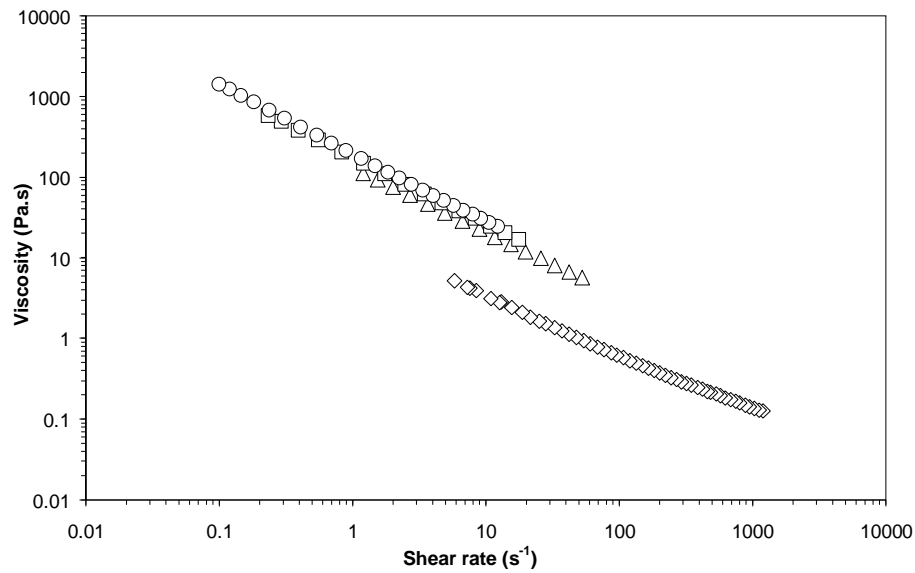


Fig.4: Viscosity curves for 0.1% ( $\diamond$ ), 0.5% ( $\Delta$ ), 1.0% ( $\square$ ) and 1.5% ( $\circ$ ) Carbopol samples

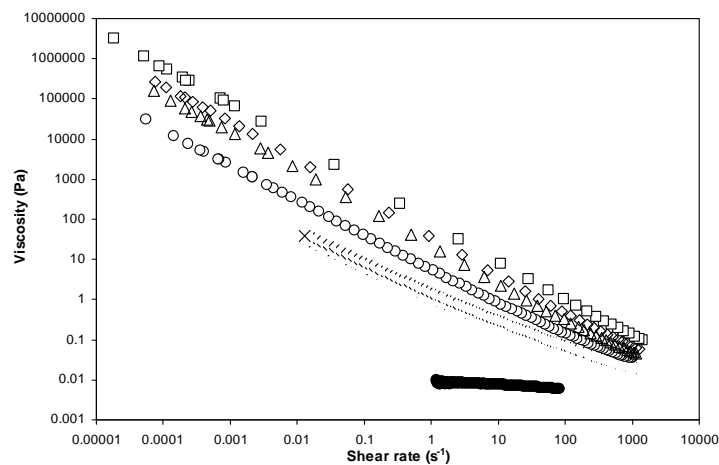


Fig. 5: Viscosity curve for different concentration of Laponite: 1.0% (●), 2.0% (X), 2.5% (○), 3.0% (Δ), 3.5% (◇) and 4.5 % (□)

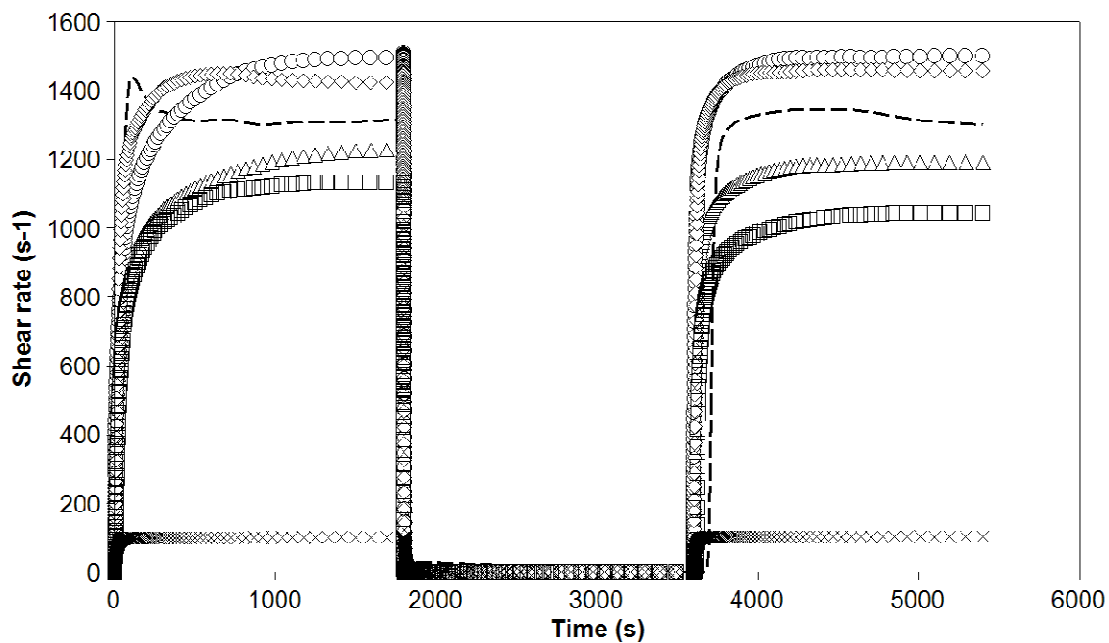
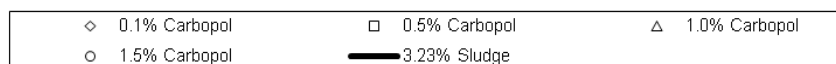
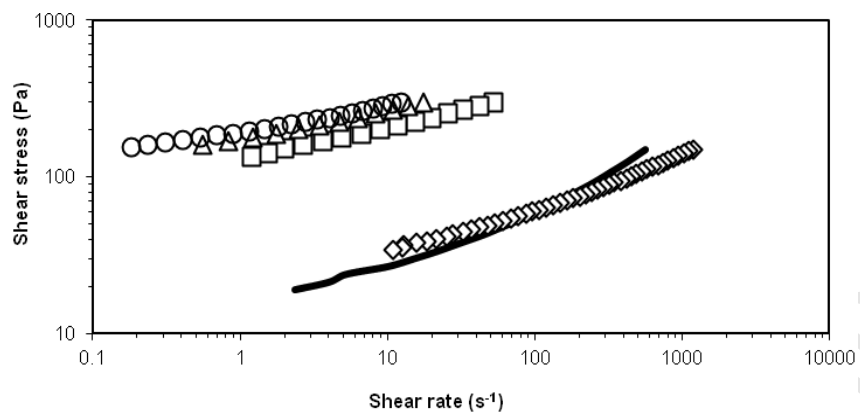
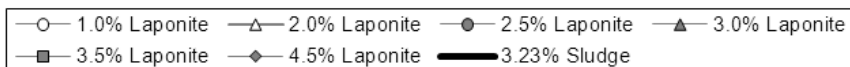
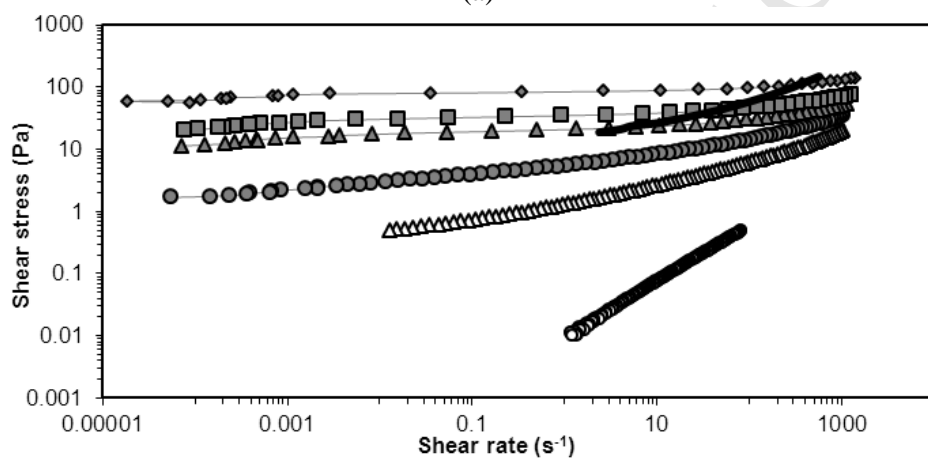


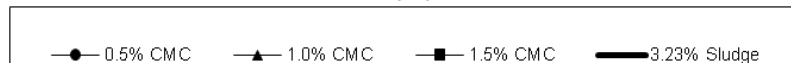
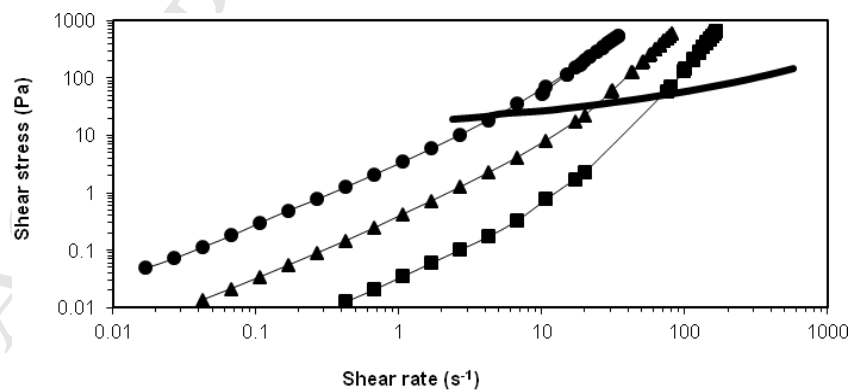
Fig.6: Thixotropic behaviour of Laponite with 30 minutes rest time between each shear: 1.0% (X), 2.0% (Δ), 2.5% (○), 3.0(◇) and 3.5 % (□) and 4.5 % (--)



(a)



(b)



(c)

Fig.7: Comparative flow curves of all model fluids a) Carbopol, b) Laponite and c) CMC) with 3.23% digested sludge

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