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EXTRUSION OF ECC: RECENT DEVELOPMENTS AND APPLICATIONS

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Abstract
Extrusion of particulate pastes and suspensions in general is difficult and the rheological parameters play a central role in the process when using conventional extruders. More important – the rheological properties of the paste or suspension are subjected to conflicting demands in an extrusion process. Extrusion of cementitious (fiber reinforced) materials has proven particularly difficult due to the high inter-particle friction combined with the disastrous effect of static zones in the flow pattern, and to the ease of phase migration or separation. In order to deal with these conflicting demands on the rheological properties of cementitious particulate materials, various methods have been suggested to dewater the particle suspension during extrusion, however practical extrusion of thin-walled cementitious large-scale elements has not been possible until the discovery of the “dewatering extrusion principle” which enabled the contradictory requirements on rheological properties to be accommodated. The present paper describes the fundamentals of the process and preferred embodiments of the extruder principle. Further the paper describes the current understanding of the dewatering process and how it can be utilized to control the extrusion process. Finally, an application of the extrusion principle is described in the shape of a thin-walled, semi-flexible ECC pipe.

1. INTRODUCTION
Two basic, conventional extruder types exist for the extrusion of particulate materials in the form of a pastes or suspensions (including fiber reinforced, cementitious materials). These are ram extruders (batch type extruders) and screw conveying extruders (continuous extruders). In each case the downstream end of the extruder consists of a die with a die entry upstream from a die land. The die entry shapes the material being extruded to the desired cross-sectional shape and further provides a cross-sectional reduction to allow the shaping process to take place under a suitable pressure.

The ram extruder is characterized by the fact that it applies pressure to a predetermined amount of material in a feed chamber by means of a piston (ram). The feed chamber leads to
the die, which shapes the material to the final extrudate geometry as it passes the die. The extrusion pressure is determined by the resistance exerted by the die on the particulate paste or suspension. That is, the extrusion pressure is determined by the combination of rheological properties of the particulate paste or suspension, the rate of movement of the piston and the die geometry (in particular the cross sectional reduction and the length over which it takes place).

In a screw conveying extruder, the piston of a ram extruder is replaced by a screw in a housing. The housing is connected to the feed chamber in such a way that the material to form the extrudate continuously can be brought under pressure and fed to the die by the screw. This process only works if the resistance to material flow backwards through the screw is greater than the resistance to material flow through the die. If this condition is fulfilled, the situation is the same as in the ram extruder in the sense that the extrusion pressure is determined by the combination of rheological properties of the particulate paste or suspension, the die geometry and the rate of extrusion – in this case determined by the rotational speed and geometry of the screw.

It follows from the above that the rheological parameters of the material being extruded play a central role in the extrusion process using conventional extruders. More important – the rheological properties of the suspension are subjected to conflicting demands in an extrusion process. Using a Bingham visco-plastic material description it can be said that dimensional stability of the extrudate requires primarily a high yield stress and high viscosity of the particulate paste or suspension (the latter requirement being particularly important if post processing is slow). In order to prevent large scale movement of the liquid phase relative to the solid particles, i.e. phase migration or separation, the viscosity of the liquid should be high (thus giving rise to high viscosity of the suspension). However, from the point of view of the flow in the extruder, the yield stress should be limited in order to prevent formation of static zones in the flow pattern of the material being extruded and to limit the extrusion pressure, while the viscosity should be limited to lubricate the process and thus limit extrusion pressure. Extrusion of cementitious (fiber reinforced) materials has proven particularly difficult due to the high inter-particle friction (resulting in high yield stress and viscosity) combined with the disastrous effect of static zones in the flow pattern due to setting and hydration, and to the ease of phase migration or separation. The relatively high self-weight of the cementitious extrudate puts high demands on the yield stress in order to maintain shape stability, which makes it even more difficult to avoid static zones in the flow pattern.

In order to deal with these conflicting demands on the rheological properties of cementitious particulate materials, especially the conflicting demands put on the yield stress, various methods have been suggested to dewater the particle suspension during extrusion (see e.g. [1] and [2]) and in this way allow for low yield stress during flow and shaping of material and a higher yield stress in the processed extrudate – thus utilizing the strong relationship between particle volume concentration and yield stress observed in such particulate pastes or suspensions [3]. However the practical extrusion of thin-walled cementitious large-scale elements has not been possible until the discovery of the dewatering extrusion principle ([4] and [5]), which enabled the contradictory requirements on the yield stress to be accommodated.

Since the original discovery of the dewatering extrusion principle significant progress has been made in gaining understanding of the basic, governing mechanisms as well as the industrial potential of the process. The present paper describes the fundamentals of the
process and preferred embodiments of the extruder principle. Further the paper describes the current understanding of the dewatering process and how it can be utilized to control the extrusion process. Finally an application of the extrusion principle is described in the shape of a thin-walled, semi-flexible ECC pipe (see e.g. [6] and [7]) which has been produced under industrial conditions and tested in full scale installations.

2. DEWATERING EXTRUSION

The so-called dewatering extrusion is different from the above mentioned conventional extrusion methods, and the conflicting demands on the rheological properties of the particulate suspension is removed by allowing excess water to be removed during extrusion. Further in contradiction to other proposed dewatering extrusion techniques, e.g. [1] and [2], in dewatering extrusion the liquid is removed under high pressure, typically 100-150 bar. The apparatus and method can be described as follows:

1. The material being extruded is initially a cementitious, fiber reinforced particle suspension, i.e. an oversaturated fiber cement paste with particles and fibers suspended in a liquid consisting of water and additives dissolved in the water. This suspension is introduced into the extrusion chamber and pressurized by a piston. Thus the back-end of the extruder is essentially a batch type ram-extruder.

2. During extrusion the material passes through the die entry which typically consists of a tapered section which allows for a certain cross-sectional reduction. It is essential that this cross sectional reduction is designed to prevent the formation of static zones in the flow pattern.

3. Downstream from the die entry the die land with constant cross section is positioned. The die land consists of three sections; upstream an inlet section. Then follows the dewatering section and finally the frictional section. The dewatering section comprises slits or openings in the die walls while the downstream frictional section has solid walls. At the dewatering section the liquid in the suspension will leave the material being extruded. The solid particles and fibers are left behind like a filter cake in a separation process. The driving pressure for this separation process is effectively the liquid pressure which again corresponds to the extrusion pressure since the material being extruded is a particle suspension. The filter cake of the separation process eventually constitutes the extrudate. Since the extrudate form a pug in the downstream end of the extruder the extrudate is further consolidated by the extrusion pressure.

4. The extrudate is held in place by the static wall friction acting against the frictional section of the die. This friction is generated by the pressure from the expansion of the extrudate, which in turn is generated by the extrusion pressure acting on the extrudate. In order to extract the extrudate, the inner or outer walls of the die are made to move relative to each other in a reciprocating manner: in a forward (downstream) stroke the extrudate follows the moving wall and in a backwards stroke the extrudate sits with the static wall because of the incompressibility of the particulate suspension in the upstream direction.

In conclusion it can be said that the high pressure separation process combined with the utilization of mechanical, static friction for allowing build-up of extrusion pressure and reciprocating die walls to extract the extrudate are unique features which distinguish
dewatering extrusion from other types of extrusion. In particular the high consolidation pressure allows formation of a very stable green fiber reinforced cementitious extrudate and very thin walled products can be produced as it will be shown in the following. Further it was shown in [8] that the extensive consolidation results in improvement of the fiber-matrix bond in typical fiber reinforced cementitious composites and thus potential for improvement of the mechanical properties of the hardened product.

2.1 In-line extrusion

A preferred geometry for a pipe extruder according to the dewatering principle is shown in Figure 1. It was found that the geometry of a previously proposed laboratory extruder [7] was not suitable for an industrial extruder, primarily due to static zones in the flow pattern and subsequent pre-consolidation in the extrusion chamber. Consequently a so-called inline principle was proposed [9] defining a batch-type ram extruder combining low pressure filling with axial high pressure flow, shaping and subsequent dewatering.

![Figure 1: The main elements of a dewatering extruder for pipe products according to the dewatering principle.](image)

The figure shows the cylindrical extrusion chamber which contains a static core and an annular piston. The cementitious particulate suspension is feed into the extrusion chamber through inlet ports at low pressure at the same time as the piston is pulled back. During extrusion the piston pressurizes the particulate suspension in a forward movement to about 100–150 bar. The particulate suspension passes though the die entry which consists of a tapered section with a cross sectional reduction onto the die land which has constant cross section. The die land comprises the dewatering section (upstream) and the friction section (downstream). The outer wall of the die consists of a reciprocating cylindrical sleeve which again contains a dewatering section and a frictional section. The frequency, \( f \), and length of the reciprocating movement, \( m \), determines the average extrusion rate, \( V \):

\[
V = \frac{mf}{2}
\]  

(1)
2.2 Steady State Extrusion

When controlling and operating a dewatering extruder it is essential that the extrudate has uniform properties. In particular it is essential that the dewatering in the extrudate is uniform. This is achieved by allowing the extrudate to grow upstream from the dewatering section of the die in order to allow that part of the extrudate to work as a filter cake. During extrusion the average extrusion rate should be adjusted to the growth rate of the dewatered extrudate – the consolidated material or filter cake. Fortunately, under certain conditions described below, the length of the filter cake is automatically adjusted to the extrusion rate.

Figure 2: Section of the extruder die land. To the right the inlet section with a constant cross section and length $L'$ is shown. The dewatering section with the extruder wall perforations is shown in the middle while the downstream frictional section is shown to the left. The consolidated material stretches $L$ past the dewatering section in the upstream direction, forming a filter cake.

The flow of water $Q$ through the filter cake is, according to Darcy’s law, proportional to the cross sectional area, $A$, the pressure drop over the filter cake (i.e. the liquid pressure, i.e. the extrusion pressure), $P$, and inverse proportional to the length of the filter cake, $L$, see also Figure 2.:

$$Q = KA \frac{P}{L'},$$

(2)

where $K$ is a constant, which according to the so-called Blake-Kozeny relationship can be interpreted as a function of the viscosity of the liquid, size distribution of the particles and the void volume fraction (or particle packing). Introducing the volume fraction of liquid being drained from the consolidated material, $w$, the following relation holds in a time step $dt$:

$$Qdt = wAdL$$

(3)
Combining Eqs. (2) and (3) results in a differential equation which solved under the condition of constant pressure results in the following relation between filter cake length and time:

\[ L(t) = \sqrt{Ct} \]  

(4)

The constant \( C \) is called the consolidation constant. The consolidation constant depends linearly on the extrusion pressure \( P \) and the constant \( K \) and inversely proportional on \( w \). The rate of growth of the filter cake can be written as:

\[ \frac{dL}{dt} = \frac{C}{2L} = \sqrt{C} \frac{1}{2\sqrt{t}} , \]

(5)

from which it follows that the growth rate slows down inversely proportional to the length of the filter cake. In a steady state situation the average extrusion rate (determined by the reciprocating movement of the dye wall) shall be equal to the average growth rate of the filter cake:

\[ V = \frac{dL}{dt} \]

(6)

Thus it follows that the length of the filter cake will automatically adjust as long as the extrusion rate is adjusted between upper and lower bounds:

\[ \Phi < V < \Theta \]

(7)

where:

\[ \Phi \propto \frac{C}{L'} = \frac{2K}{w} \frac{P}{L'} \quad \text{and} \quad \Theta \propto \frac{C}{T} = \frac{2K}{w} \frac{P}{T} \]

(8)

Here \( L' \) is the maximum allowable length of the filter cake, corresponding to the length of the die inlet section upstream from the dewatering section. (Growth beyond that point into the tapered section will lead to blockage of the extruder, since the consolidated material is almost un-deformable.) According to Eq. (5) there is no upper limit for the filter cake growth rate, however in practice a filter cake thickness smaller than the extrudate wall thickness, \( T \), will never be allowed in order to maintain the situation outlined in Figure 2. This condition is reflected in the upper bound in Eq. (7). In practice upper and lower bounds for the extrusion rate of a given material in a given specific dewatering extruder will always have to be determined empirically, however Eqs. (7) and (8) give valuable information about the dominating parametric dependencies. See also [10].

It follows from the above that the consolidation constant \( C \) contains key information about how efficient a given fiber reinforced suspension can be extruded. The consolidation constant \( C \) can be measured from a simple consolidation experiment [10] and should be on a regular basis during production.
Using a pilot extruder extruding 375mm pipe with 12mm wall thickness at an extrusion pressure of 10MPa fiber reinforced cementitious particulate suspensions with different consolidation constants were extruded at various extrusion rates and the stability of the process observed. The results are plotted in Figure 4 indicating also the experimentally observed linear relationship between $C$ and maximum extrusion rate obtained by fitting a straight line between stable and unstable extrusion rates. Also the calculated, minimum extrusion rate is shown based on an inlet length of 100 mm.

3. SEMI-FLEXIBLE EXTRUDED ECC PIPE

The process principles described above were utilized in setting up a pilot plant for production of 375mm pipe with 12mm wall thickness in sections of 4m length for underground use. The material used was a polypropylene (pp) fiber reinforced cementitious ECC material with a matrix consisting of Portland cement, fly ash and particulate material in the form of quarts powder and fine sand. The pseudo strain hardening (p.s.h.) behavior or the ECC material allowed for multiple cracking behavior in bending which, together with a suitable choice of wall thickness, gave the behavior indicated in Figure 5 when sections of the pipe were subjected to diametrical, quasi-static bending in the three point test setup prescribed in standards, e.g. AS 4139-2003.

The behavior is characterized though an equivalent stress versus relative displacement diagram where the equivalent stress, $\sigma_e$ is defined by:
\[
\sigma_e = k \frac{6}{\pi} \frac{p}{D_y} \frac{1 + \frac{D}{D_y}}{\left(1 - \frac{D}{D_y}\right)^2},
\]  

(10)

where \(k\) is a close to unity factor taking into account the distance between the two bottom supporting edges, \(p\) is the line load intensity while \(D\) and \(D_y\) designate the inner and outer diameter respectively and where the relative displacement is measured absolute displacement relative to the inner diameter. Referring to Figure 5, the behavior can be divided into a linear part ending at stress LOP corresponding to a relative deformation \(\delta_1\), a first strain hardening part where a pseudo plastic regime is developed on the inside of the pipe in the vertical symmetry plane ending at relative deformation \(\delta_2\) and a second part where a pseudo plastic regime is developed on the outside of the pipe in the horizontal symmetry plane ending at stress MOR. A particularly favorable pipe behavior, here defined as semi-flexible, is obtained when LOP is about 5-7 MPa, corresponding to a relative displacement \(\delta_1\) of 0.6 to 0.8%, when \(\delta_2\) corresponds to about 1.2% and when MOR is about 11 to 15 MPa corresponding to a relative deformation \(\delta_3\) of 5 to 9%. The characteristic slopes \(S_1\) and \(S_3\) follow from these quantities. See also [11].

Figure 5: Typical effective stress versus relative displacement response of a pseudo strain hardening, thin walled, semi-flexible pipe in standard test configuration. The extent of the multiples cracking regimes is indicated to the right in p.s.h. part (I) and (II).

Figure 7 shows engineered trial installations of the extruded semi-flexible pipe under an access road to a quarry, where the pipes were loaded from the heavy trucks. The pipes behaved satisfactorily and showed deflections of about 0.7% after 130 hours of intermittent loading indicating the preferred behavior mode where dead load alone loads the pipe below
LOP while peak service loads activate the first part of the pseudo strain hardening behavior. Laboratory testing indicated a service life beyond 10 mill. cycles under these conditions.

**Figure 6**: Figure to the left is showing 12mm Ø 375mm pipe in 4m long sections. The pipes are extruded pp-ECC semi-flexible pipe. The picture to the right is showing the installation procedure.

**Figure 7**: Overview of the pilot plan.

An overview of the pilot plant for 375mm pipe production is shown on Figure 7. The extruder and feeding arrangement is shown in the bottom left. Pipes are extruded onto troughs.
which are subsequently brought into a steam curing chamber (top center) on a conveyer line (center). The computer for operational control is seen just to the left of the entrance to the steam curing house. Pipes are stacked after steam curing. To the far right empty troughs are waiting to be filled.

11. CONCLUSIONS

− A new extrusion principle called dewatering extrusion, suitable for extrusion of fiber reinforced cementitious composites, has been demonstrated full scale in the production of 375 mm thin walled (12 mm) pipe in 4 m sections.
− Principles for preferred designs of the extrusion machine have been laid out. These principles include low pressure filling of the extrusion chamber and high pressure extrusion involving only axially symmetric flow. The die contains a dewatering section and a frictional section where the particle suspension is dewatered and where the consolidated material is subsequently held in place as a plug by wall friction allowing the build up of the extrusion pressure. The consolidated material or the extrudate is extracted by a reciprocating movement of the outer die wall.
− Principles for so-called steady state operation of the extruder have been laid out and quantified. These principles explain the basic relationship between extrusion pressure, material composition and maximum attainable extrusion rate.
− A new semi-flexible fiber reinforced cementitious pipe was designed utilizing ECC. The pseudo strain hardening of an ECC material provides flexibility for peak loading as well as suitable safety for both fatigue load and static overload.

REFERENCES