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Characterisation of the wall-slip during extrusion of heavy-clay products

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Abstract. During extrusion through the extrusion die, heavy-clay compounds are usually show plug flow with extensive slip at the wall of the die. In this study, the viscosity and the thickness of the slip layer were investigated. For the examination a brick-clay from Malyi (Hungary) deposit was applied as a raw material. The clay was characterised by XRPD, BET, SEM and granulometry. As the slip layer consists of suspension of the fine clay fraction so the clay minerals content of the clay ($d < 2\mu\text{m}$) was separated by the help of sedimentation. The viscosity of suspension with different water content was measured by means of rotational viscosimeter. The thickness of the slip layer was calculated from the measured viscosity and other data obtained from an earlier study with capillary rheometer. The calculated thickness value showed a tendency to reach a limit value by increasing the extrusion speed.

1. Introduction

The production of the traditional fired brick product demands the preparation and shaping of the largest volume of plastic ceramic pastes. Additive materials are often mixed into the clay based compounds in order to change an unbeneficial behaviour of the clay (i.g. too high plasticity) or to improve the properties of the fired products [1-5]. Results from rheological and tribological studies established that not only the clay but the clay-compounds can also be described as Bingham-fluid at low shear rate [6,7,8]. However it was also observed that a thin layer built up from clay-suspension between the forming die and the clay body [9]. Accordingly the extrusion behaviour of the these materials is influenced by the slip layer beside the shear stress. The aim of this study is the examination of the layer and the calculation of the layer thickness based on former studies.

2. Background

During extrusion the clay-water suspension fills up the void space between clay particles and owing to the rapidly increasing pressure it flows in the direction of the die wall [6, 10]. The rigid particles of the clay behaves as a filter and it retain the rougher fraction of the suspension so a thin layer builds up on the surface of the extrudate (or on the die wall if we approach from the other side) which has its own viscosity and thickness. This two behaviours are the function of more parameters such as the composition of the clay; the extrusion pressure and surface roughness [11]. In the extrusion process the layer affects the volume of the slip and share fraction of the extruded body. A typical flow of a clay or a clay based compounds can be seen on the figure 1. The liquid layer at the wall developed not only by clay-water systems but the at the production of technical ceramic pastes [12-14], drugs [15] products of the food industry [16], polymers [17] or at the flow of foams, cements through a pipe [18]. For the examination of the layer beside the rheological methods technique based on magnetic



resonance are also applied [19]. The determination of the rheological parameters and wall slip based on shaping technology demands a sophisticated method like capillary rheometry [11]. From such a measurement the true flow curve can be obtained with the description of the flow behaviour.

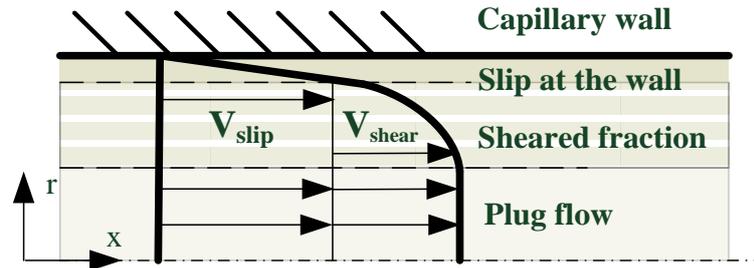


Figure 1. Generalized capillary flow profiles of the clay or clay-based compounds

The development of the slip layer is the function of the rheological behaviour of the liquid phase, particle morphology and size distribution of the clay or ceramic pastes [15]. For their alumina paste, Gracyk and Gleissle [20, 21] found that although the flow through the capillary was entirely plug flow, there was a dependency of the wall shear stress upon the diameter of the capillary. As thickness of the slip layer compared to the diameter of the capillary is negligible, it was supposed that the slipping speed (v_{slip}) and the thickness of the layer (s) is linearly proportional as follows:

$$\frac{v_{slip}}{s} \approx \dot{\gamma}_s \quad (1) \quad \text{and} \quad \frac{\tau}{\dot{\gamma}_s} = \eta_s \quad (2)$$

where $\dot{\gamma}_s$ is the shear rate in the layer, η_s is the viscosity of the layer and τ is the wall shear stress.

Equating the two expression Eq. (1) and Eq. (2), a quantity was introduced: reference slip layer thickness: s_r (Eq.3) Higher s_r value means an increase in s and decreases in η_s .

$$s_r = \frac{s}{\eta_s} = \frac{v_{slip}}{\tau} \quad (3)$$

s_r is easily calculable as v_{slip} and τ are obtainable from a measurement with a capillary rheometer. The method was applied by Khan et al [22] for the measurement of alumina paste.

According to Eq. 2 the real thickness of the slip layer (s) remained unknown. Supposing that the value η_s from the Eq.3 is measurable then the real layer thickness becomes calculable because the shear stress and the slip velocity is available [8]. Based on former experiments [8, 10] the following conditions were assumed:

- the slip layer build up from a suspension of water and the $d < 2 \mu\text{m}$ component of the clay;
- the moisture content of the layer changes according to the clay mixture;
- the thickness of the layer increases as the pressure increases;
- slipping speed (v_{slip}) and the thickness of the layer (s) is linearly proportional.

Some related studies supposed some μm for thickness of the layer. Applying Eq. (3) and supposing 1 μm and 5 μm for the thickness of the slip layer, the calculation with $v_{slip} = 100\text{mm/s}$ data from measurements with capillary rheometer [8] resulted the shear rate in the layer:

$$\begin{array}{l} s_1 = 1 \mu\text{m} \\ s_2 = 5 \mu\text{m} \end{array} \quad \longrightarrow \quad \begin{array}{l} \dot{\gamma}_{s1} = 100/0,001 = 100000 \text{ 1/s} \\ \dot{\gamma}_{s2} = 100/0,005 = 20000 \text{ 1/s.} \end{array}$$

Consequently the shear rate can be as high as 100000 1/s in the layer assuming Newtonian fluid. Higher shear rates usually measure with capillary rheometer but it is non usable for clay-suspension. Applying rotational viscometer the measuring limit can be elevated over 1000 1/s and the behaviour of the suspension became observable.

3. Materials and methods

An ordinary brick clay with high plasticity (Atterberg number: 21.5) from the Mályi clay deposit (Hungary) was chosen as the raw material for the examinations. Before preparation of the material, it was characterised by the XPRD, BET, granulometry and morphology (Table 1.). The mineral composition of the yellow clay was analysed by XRPD applying a Bruker D8 Advance X-ray diffractometer. The quantitative result was calculated by Rietveld profile refinement. The yellow clay consisted of 35.2% clay minerals. Roentgen amorphous materials could be considered to be plastic components in the clay because these materials are very fine graded aluminium- and iron-oxide-hydroxides. Oligoclase and microcline represented the feldspar content in an amount of 9.8%. In addition to the 31.1% free quartz, there were a small amount of dolomite and calcite. Because of the clay minerals and relatively high amount of roentgen amorphous materials, the clay body showed a highly plastic and sticking behaviour. The mica content was 15.3%. The particle size distribution was determined by Horiba LA950 laser granulometer and the specific surface was measured on a Micrometrics Tristar 3000 instrument. In order to get the finest particle of the clay, the particles were separated by sedimentation into seven fractions including: $5\mu\text{m} < d < 15\mu\text{m}$; $2\mu\text{m} < d < 5\mu\text{m}$; $d < 2\mu\text{m}$. Analysing morphology by means of a Hitachi TM-1000 tabletop SEM found the particles of the clay have mainly anisotropic shape. The morphology of two fraction can be seen on the Fig 2.

Table 1. Mineralogical composition (wt%), grain size distribution and specific surface area BET surface area of the clay

Oligoclase	Microcline	Calcite	Dolomite	Quartz	Mica	Illite	Montmorillonite	Kaolinite	XRD amorphous
5,4%	4,4%	1,2%	0,9%	31,1%	15,3%	23,1%	7,9%	4,2%	6,4%
Granulometry (μm)	$d_{0,1}$	$d_{0,5}$	$d_{0,9}$	BET in fraction (μm)		$d < 2$	$2 < d < 5$	$5 < d < 15$	$15 < d < 45$
	1,9	11,9	50,8	(m ² /g)		~37	~48	~34	~15

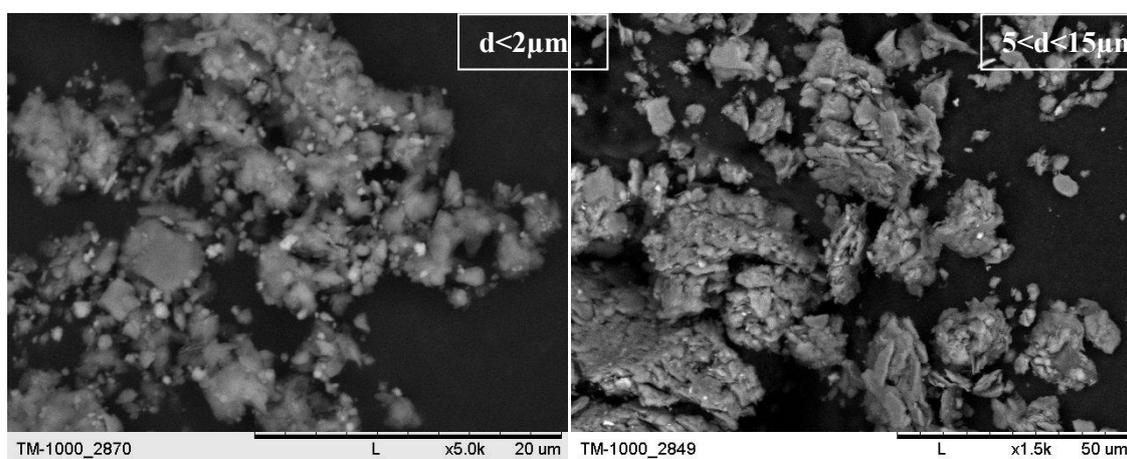


Figure 2. Morphology of the clay fractions

According to the above declared conditions, two clay suspension with different moisture content was prepared. For the examination, the fraction of $d < 2\mu\text{m}$ was applied and the relative moisture content was adjusted to $w=28\%$ and $w=50\%$ by adding water to the suspension. The mixture with lower moisture content was highly plastic while the other was relatively fluent. The rheological tests were carried out on a rotational viscometer (Fig.3. Haake RS80; head: CP 2Ti with 25mm diameter). The gap was set to 0,1mm and the ambient temperature was 23°C. During measurement slow ramp time was applied up to the maximal shear rate of 1600 1/s.



Figure 3. Viscometer RS80 and the CP 2Ti 25mm measure head

4. Results and Discussion

The results of the measurements were the flow curves of the suspensions. Each test was carried out three times and an average of viscosity value was calculated. The shear stress-shear rate and viscosity curves are presented on the Fig. 4. The Fig 5 represents the average stress and viscosity values.

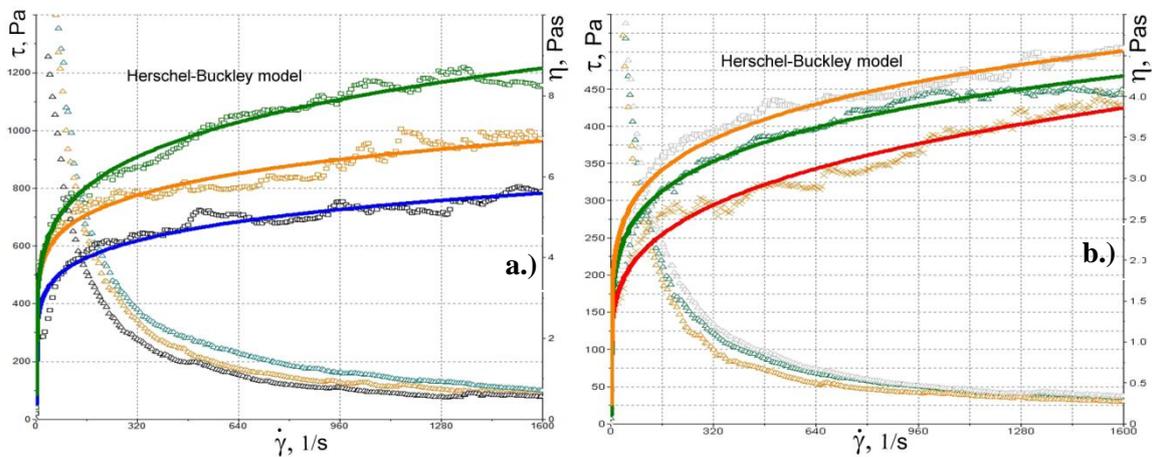


Figure 4. Flow curves for fraction below 2 μm, moisture content w=28% (a.) and w=50%-os (b.)

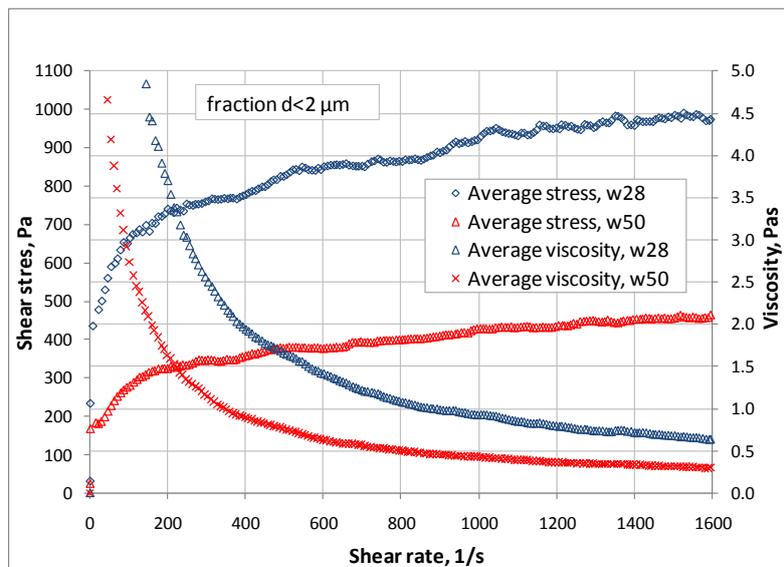


Figure 5. Averaged flow curves for fraction below 2 μm, moisture content w=28% (a.) and w=50%-os (b.)

The viscosity results are summarized in the Table 2. It is visible in Fig 5 that as the shear rate increases the viscosity of the suspension tends to reach a limit value. The viscosity of the suspension was below 1 Pas for both moisture content. It follows from this that the viscosity of the slip layer can be changed between the viscosity of the pure liquid phase (viscosity of water is 0.001 Pas) and around 0,64 Pas. Supposing that the non-compressibility of the suspension and take the viscosity value as constant at high shear rates, it can be extended over 10000 1/s. Results also show that the clay fraction ($d < 2 \mu\text{m}$) followed the Herschel-Buckley model (Eq.4) for both water content. The model parameter and coefficient of determination are summarized in table 3.

$$\tau = \tau_0 + K\dot{\gamma}^n \quad (4)$$

Table 2. Viscosity of clay-water suspension

Fraction	Water content w, %	Viscosity over 1000 1/s, $\dot{\eta}$, Pas
$d < 2 \mu\text{m}$	28	0,64
$d < 2 \mu\text{m}$	50	0,27

Table 3. Model parameters

	τ_0 Pa	K Pas	n	r^2
w=28%	76,20	259,40	0,166	0,94
w=50%	37,74	96,22	0,179	0,96

Applying the viscosity value in Eq.3, the “s” layer thickness was calculated. The table 4 shows the values of the shear stress and the slip velocity which were the basis of the calculation. They were obtained from the analysis of the same yellow clay with capillary rheometer and they were presented in other study [8]. The ram speed affected the slip velocity and the shear stress for all applied capillary diameter ($d=3; 4$ and 5mm). The Fig.6 illustrates the calculated slip layer thickness as a function of extrusion (ram) speed for three different capillary diameter. During shaping, the calculated thickness of the slip layer resulted in some micrometer.

Table 4. The shear stress and the slip velocity data for different capillary

τ Pa	$v_{cs\ d3}$ mm/s	$v_{cs\ d4}$ mm/s	$v_{cs\ d5}$ mm/s
0,27	25,32	4,69	1,79
0,29	35,57	9,23	4,28
0,31	46,42	13,55	6,74
0,33	57,40	17,82	9,18
0,35	68,41	22,06	11,61
0,37	79,45	26,28	14,04
0,39	90,50	30,50	16,47
0,41	101,56	34,71	18,90

During extrusion of clay and clay-based compounds the layer behaves similar to this. Fig. 6 shows that the layer was also thicker as the extrusion speed increased. As extrusion speed and the extrusion pressure increase parallel it facilitates the flow of the suspension toward the die wall. The thickness is tends to reach a limit and this limit value is the function of the mineral and water content and also the extrusion pressure and surface roughness. Former studies proved [6, 7, 8] that clay and the clay with additives (clay compounds with pore former and opening agents) behaves similar i.e. Bingham model. However the thickness of the layer was calculated point to point from the measurement data and it showed non-linear tendency.

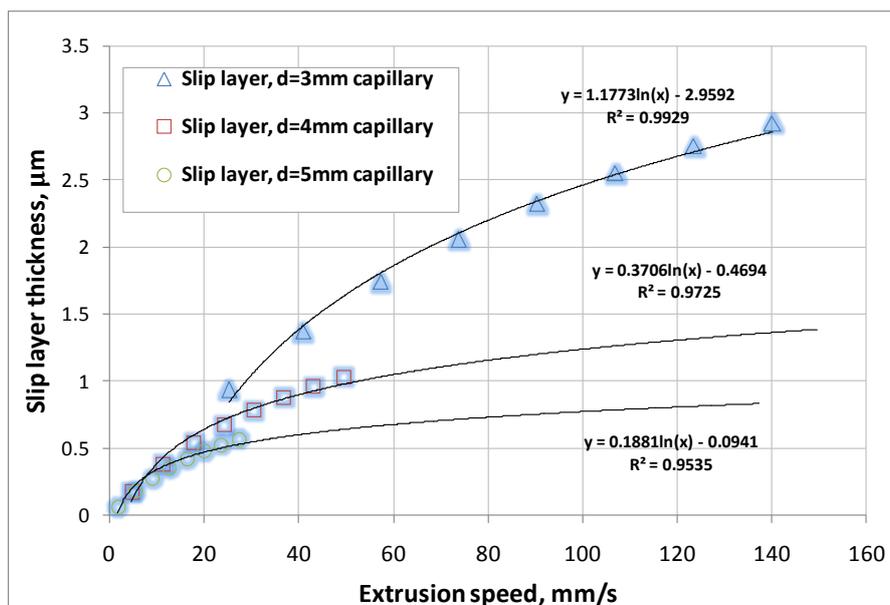


Figure 6. The change of thickness of the slip layer as the function of extrusion speed

Summary

In this study the slip layer was examined which is usually built on the surface of a heavy-clay products during shaping. The layer is composed of suspension of the finest clay particles and water. The basic condition for formation of the layer was defined and it was characterised by means of capillary and rotational rheometer. The viscosity of the clay fraction ($d < 2\mu\text{m}$) was determined and based on the results of earlier tests of the clay the layer thickness was calculated..

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