Significance of an inclined plane test for mould slag assessment.

I. Marschall, H. Harmuth, V. Kircher

The viscosity of mould slags is one key parameter in the continuous casting process of steel. Its measurement using a viscometer is costly and time and consuming. Therefore, beside viscosity modelling, an inclined plane test to determine viscosity was proposed by several authors. The task of this study was to find out, whether this method is suitable for quality control of mould slags, or not. Therefore, an inclined plane test device (IPT) was constructed and operated. At the same time, the slags were investigated by a viscometer. In total, 80 different slag compositions were investigated. Several options to represent the viscosity as a function of the ribbon length have been studied, and a suitable relation is proposed. It serves for detailed statistical studies to assess the error of IPT. While the relation between viscosity and length shows a satisfactory degree of determination (e.g.>0.97) in this study, this still does not ensure sufficient accuracy of individual applications of IPT. As the slag undergoes cooling during the test, also the viscosity change in dependence of the temperature contributes to the result, not only the viscosity of the initial impact temperature. This raises the unexplained variance of the result.

KEYWORDS: MOULD SLAG, MOULD FLUX , VISCOSITY, INCLINED PLANE TEST. RIBBON LENGHTH

BACKGROUND

Viscosity is an important physical property of mould slags used in the continuous casting process of steel since it controls the infiltration and the lubricity in the gap between the mould and the steel strand. It influences the crystallisation behaviour and therefore the heat flux from the steel to the mould wall. Furthermore, the viscosity is involved in the absorption of non-metallic inclusions from the steel and it influences their dissolution. Viscosity usually decreases with increasing temperature and depends on the chemical composition of the casting powder. A fast and easy estimation of viscosities on site is desired from steel makers.

The inclined plane test (IPT) seems to be an easy method for this purpose. Based on the approach of the modified Herty viscometer [1], viscosities of slags are determined by the length of a slag ribbon formed. At Hertys device, the slags solidified in a horizontal steel tube, whereas Mills et al. [2] performed their experiments on a steel plane and showed that the ribbon length is a function of the inclination of the plane. Due to the fact that for more viscous slags the ribbon lengths decreased, they adjusted the inclination from 9 ± 0.5° up to 23° to obtain longer ribbons. Brandaleze et al. [3] adopted it as a quick method

Irmtraud Marschall
K1-MET GmbH, Austria

Harald Harmuth
Montanuniversität Leoben, Austria

Volkmar Kircher
Montanuniversität Leoben, Austria

to measure the viscosity/fluidity of mould powders being used for continuous casting of different steel qualities. While previous studies [2,4] were performed focusing mainly on mould slags with viscosities >0.15 Pa.s at 1300 °C and fayalite slags at 1480 °C [5], in the present study the mould slag viscosity of 62 % of the samples is below 0.15 Pa.s at 1300 °C. Therefore, the application of this method for mould slags showing lower viscosities have been tested. The significance of IPT was assessed by the measurements of 80 different slags.

EXPERIMENTAL

Device

15 g of pre-molten slags were heated up within a platinum crucible in a muffle type furnace and the liquid melt was poured onto an inclined trough cut from a stainless steel tube with a diameter of 50 mm and a length of 645 mm. The detailed setup is given in Fig. 1. In accordance with [4], 14° was spotted to be the optimum inclination of the trough (1). Due to the fact that the slag cools down very fast at room temperature an easy to handle pouring mechanism is necessary. To standardize the slag impact on the inclined plane, a rotatable insulating crucible holder (2), manufactured out of a steel cup lined with light-weight refractory material, was installed. A trigger lever (3) ensures uniform start of casting. Additionally, a retaining device (4) fixes the Pt-crucible during tipping. Several angular velocities were tested, adjusted by balancing weights (5) and the ejection height was set to 40 mm to enable the best possible casting conditions. In case of a too low rotational speed, the slag ribbon was not continuous. If the rotational speed was too high, a first droplet was catapulted down the inclined plane without contact to the residual ribbon. Best results were obtained with a rotational speed of 3.41 rad sec^{-1}. The slag temperature was measured during the slag impact by a type-S thermocouple (6), connected to a data logger (7). Example of casted ribbons may be seen from detail 8 in Fig.1 and Fig.2.

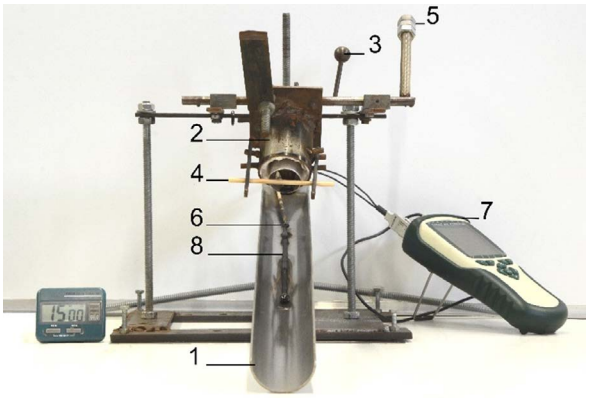

Fig.1 - Inclined plane test in operation; consisting of 1) an inclined trough, 2) a crucible holder, 3) a trigger lever, 4) a re-taining device, 5) balancing weights, 6) a type-S thermocouple, 7) a datalogger and 8) slag ribbon.

Fig.2 - a) Droplet formation in IPT ribbons of a SG5 slag according to Tab.1 with a viscosity of 0.079 Pa.s at 1300 °C; b) IPT ribbons of a glassy solidified SG5 slag with a viscosity of 0.139 Pa.s at 1300 °C, c) IPT ribbons of a SG1 slag with a viscosity at 1300 °C of 1.546 Pa.s.

Temperature measurement

As viscosity is highly dependent on temperature, it is indispensable to keep the temperature constant for ac-curate measurements. Throughout the development process, two different methods for sample temperature measurement have been used. First trials were accompanied by a thermal imaging camera. It was applied to determine the temperature drop of the slag from muffle furnace to the IPT, during casting and during solidifi-cation of the slag ribbon. In Fig.3 a), a thermal image of the temperature distribution of a solidifying slag ribbon is shown,3 seconds after the first slag drop hit the tube. In the upper part of the image, the remaining heat of the crucible can be seen. From the thin ribbon with a droplet at the upper and lower end it is obvious that slag viscosity was rather low. Thinner parts are cooling down much faster than droplets, as it can be seen in Fig.3b). Here, the cooling of the same slag ribbon is illustrated, 3 and 13 seconds after slag impact. Due to this temperature measurement method, it was possible to optimize the furnace temperature, and to fix the testing procedure with the help of thermocouple measurements as shown below. It was important to consider that the core temperature of the ribbon is higher than the surface temperature measured by thermal imaging.

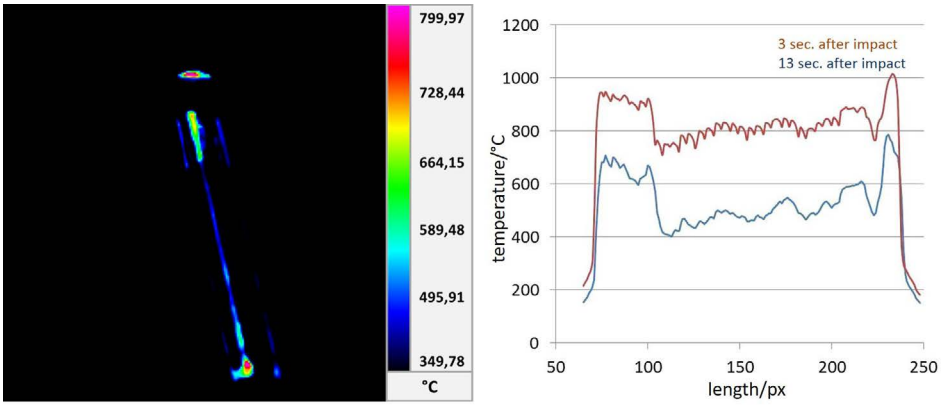

Fig.3 - a) Thermal image of the temperature distribution of a solidifying slag ribbon and b) temperature distribution dia-gram of the slag ribbon, 3 and 11 seconds after slag impact.

In a second trial, the cooling of the slag in dependence on the transfer time between furnace and the start of pouring was tested. The slag used had a CaO/SiO_2 ratio of 0.97, a measured viscosity of 0.19 Pa.s at 1300 °C and a break temperature of 1132 °C. The furnace temperature was kept constant at 1490 °C, the transfer time varied and the temperature at impact was recorded by a type S-thermocouple situated at the impact area on the inclined trough. For each time step three experiments have been performed. Form the results (see Fig. 4) follows that the transfer time has a crucial impact on the ribbon length.

For the final setup, the temperature of the slag at impact was controlled by a thermocouple installed on the inclined trough. To guarantee about 1300 °C at impact, the slag is heated up to 1490 °C with a dwell time of 15 min. The transfer time of the crucible between furnace and IPT start is fixed to 5 s. For each sample, the test was repeated five times.

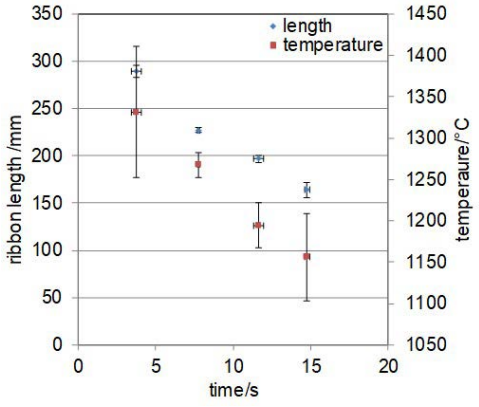

Fig.4 - Ribbon length and temperature at impact in dependence on the transfer time between furnace and tipping of the crucible.

Viscosity measurement and determination of the liquidus temperature

To correlate the IPT ribbon length with the dynamic viscosity, viscosity measurements were performed with a Bähr V403 rotational viscometer. The crucible as well as the spindle are made of platinum (FKS-Pt/Au 95/5). 27 g of pre-molten slag were heated up to 1430 °C within 60 minutes. After a dwell time of 30 minutes, the measurements were started and the slags cooled down with 10 °Cmin^{-1} until the torque reached 50 mNm. The break temperature as well as the viscosity at 1300 °C were obtained by the viscosity curve. For the determination of the liquidus temperature, a simultaneous thermal analysis was performed with a STA device Jupiter F3 from company Netzsch. A mass of 100 mg of pre-molten slag was heated up in argon atmosphere with 5 °Cmin^{-1} to at least 50 °C above liquidus temperature.

Samples

80 samples were investigated in total. Their chemical composition can be classified into 6 slag groups (SG) as shown in Tab. 1. These are fluorine free slags with substitution of CaO by SrO (SG1) and B_2O_3 (SG2), fluorine containing calcium aluminate slags (SG3), and fluorine containing slags with a CaO/SiO_2 ratio 1.2-1.4 (SG4), 0.9-1.2 (SG5) and <0.9 (SG6).

Tab.1 - Chemical compositions of slag groups (SG) investigated, wt%

component	SG1	SG2	SG3	SG4	SG5	SG6
F			11.5-11.7	7.5-8.7	4.9-9.2	6.4-8.4
$Na_2O + K_2O$	9.9-11.9	8.0-9.2	2.0	4.9-8.7	3.6-6.9	5.3-8.5
MgO	2-2.4	4.5	2.3	1.2-2.7	0.9-5.1	0.3-2.9
Al_2O_3	11.9-14.2	3.5	28.8-29.3	3.2-8.4	1.8-13.2	5.1-14.2
SiO_2	40.6-48.5	30.9-37.8	5.8-5.9	30.7-36.2	31.6-38.5	37.2-42.5
CaO	21.2-34.1	30.2-37.1	44.9-45.8	40.4-44.0	33.5-37.1	32.7-36.1
TiO_2		8.8		0-0.2	0-9.2	0-9.3
Fe_2O_3	1.6-1.9	1.0		0-1.8	0.7-2.6	1.6-2.2
additional oxides	SrO: 0-36.1	B2O3: 5.0	B_2O_3: 2.9	ZrO_2: 0-0.1	ZrO_2: 0-9.4	ZrO_2: 0-9.2
		LiO_2: 0-1.2	MnO: 0-1.9	MnO: 0-1.0		
C/S	0.4-0.8	0.8-1.2	7.7-7.8	1.2-1.4	0.9-1.2	0.8-0.9
NBO/T	0.93-1.13	1.02-1.21	0.94-0.96	1.10-1.54	1.02-1.45	0.88-1.3

RESULTS

Correlations

For each sample, the test was performed at least five times to receive representative results. After casting, the ribbon length has been measured by a ruler and the slag remaining inside the Pt-crucible has been weighted. The mean residual masses were in a range of 0.4 to 4.9 g with standard deviations of 0.02 to 0.28 g. The ribbons gauged in this work had a length in the range of 94 to 465 mm with a standard deviation of 1.5 to 19 mm. In accordance with Mills et al. [2], it could be confirmed that there is more scatter of the ribbon length results for more fluid slags and that the liquidus temperature is of no influence as long as it is above the casting temperature. The mean of all measured slag temperatures at the impact on the trough was 1309 ± 17.5 °C. Due to these rather small temperature deviations, caused by transfer time, ambient temperature and the point of impact on the thermocouple there are no indications for an impact of this scatter on the ribbon length.

Regardless of the chemical composition, all ribbon lengths derived by IPT were correlated with the viscosity. For the relationship of ribbon length and viscosity, seve-

ral researches use different approaches. Mills [2] quoted a linear relation between the ribbon length and the fluidity for mould slags with a viscosity >0.16 Pa.s. The mean viscosity in Mills study was 3.5 Pa.s. Bazan et al [5] and Riaz [4] introduced both, an exponential approach for fayalite slags equ. (1) and mould flux equ. (2) respectively. Here η is the viscosity in Pa.s and L the ribbon length in m. Numerical coefficients are recalculated for Si units.

$$\eta = 0.154 \cdot e^{-2,56 \cdot L^3} \quad [1]$$

$$\eta = 0.02656 \cdot e^{\frac{0.47}{L}} \quad [2]$$

For fayalite slags the calculated mean viscosity at 1300 °C is 0.152 Pa.s [5] and is therefore in the same range as the viscosities of the present study, but the melting temperatures were around 1460 °C, so that Bazan performed the IPT tests at 1480 °C. The mean viscosity of the mould slags investigated by Day and Riaz are in the same range as for the slags investigated by Mills. Therefore, four cases are compared in Tab- 2 for the general exponential approach equ. (3), where c_1 and c_2 are coefficients and n is the exponent of L. The limit graph of cases 1 to 4 is depicted in Fig. 5. From this follows that cases1, 3 and 4 may be more appropriate than case 2 which shows and inflection point.

$$\eta = c_1 \cdot e^{c_2 \cdot L^n} \quad [3]$$

Tab.2 - Limits of exponential function

Case nr.	c_2	n	y(L→0)	y(L→∞)	y'(L→0)	y'(L→∞)	Author
1	<0	1	1	0	c2	0	
2	<0	>1	1	0	0	0	Bazan
3	>0	<0	∞	1	-∞	0	Dey
4	<0	0<n<1	1	0	∞	0	present study

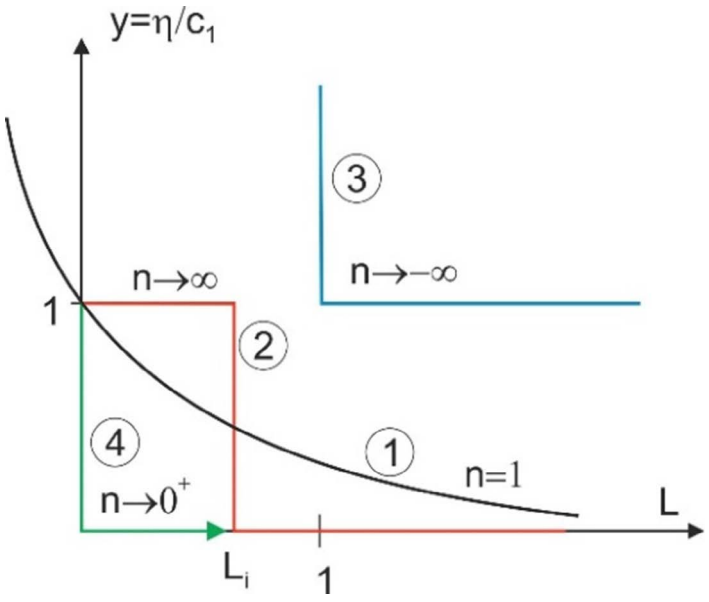

Fig.5 - Limit graph of cases 1 to 4.

Additionally, the hyperbolic approach of equ. (4) was tested with coefficients $c_1>0$ and c_2 and the exponent $n<0$. Here the viscosity will tend to ∞ for very short ribbon length and to c2 for $L\rightarrow\infty$.

$$\eta = c_1 \cdot L^n + c_2 \quad [4]$$

Results and simulations

The data given by Dey [4], Bazan [5] and the present study were evaluated by regression for the different approaches shown above. The results for the coefficient of variation of the measured, used viscosities CV(η_m), the exponent n of the length, the coefficient of determination B and the coefficient of variation of residue ε of the regression model CV(ε) are summarized in Tab. 3. For the present study, the exponential approach of equ. (5) as well as the hyperbolic approach of equ. (6) seem to be reasonable (Fig. 5). SG3 data were not considered in the regression analysis, as the ribbons show high crystallization and the values do not fit to the others. Tab. 3 shows that the values of Dey [4] fit well with his individually chosen exponent n=-1.

$$\eta = 0.0358 \cdot L^{-1.64} - 0.105 \quad [5]$$

$$\eta = 27{,}94 \cdot e^{-9.815 \cdot L^{0.523}} \quad [6]$$

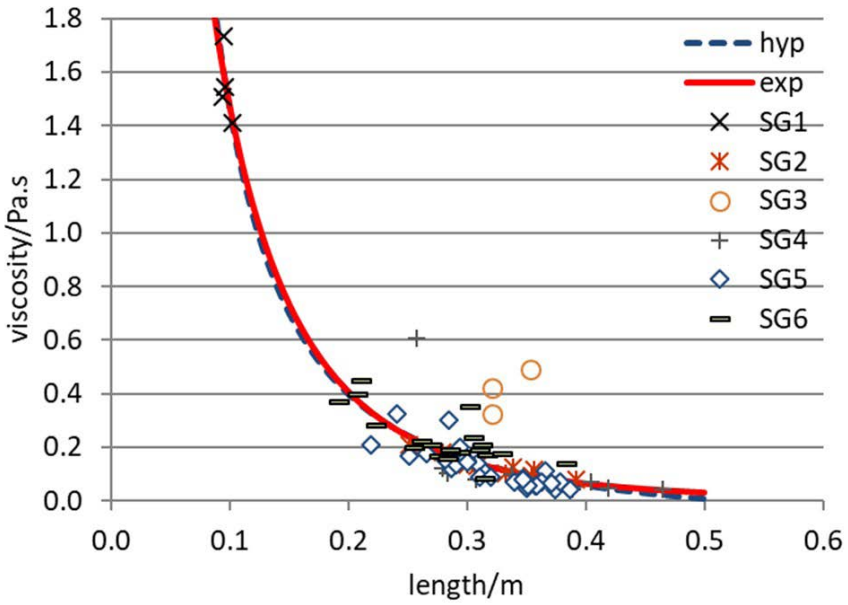

Fig.6 - Ribbon length depending on the viscosity.

Tab.3 - Results of simulations.

		Dey	Bazan	Present study
$CV(\eta_m)$		1.391	0.529	1.492
$\eta = c_1 \cdot e^{c_2 \cdot L^n}$ exponent n according to [4,5]	n	-1	3	
	B	0.9966	0.7897	
	CV(ε)	0.082	0.243	
$\eta = c_1 \cdot e^{c_2 \cdot L^n}$	n	-1.1140	9.9562	0.5234
	B	0.9967	0.8427	0.9769
	CV(ε)	0.080	0.210	0.227
$\eta = c_1 \cdot L^n$	n	-3.5723	-1.2235	-2.0452
	B	-3.5723	-1.2235	-2.0452
	CV(ε)	0.159	0.340	0.241
$\eta = c_1 \cdot L^n + c_2$	n	-4.5507	2.3758	-1.6405
	B	0.9956	0.8193	0.9771
	CV(ε)	0.093	0.225	0.226

Statistical assessment

This paragraph considers the hyperbolic approach according to equ. (4) and the results received for the present study according to Tab. 3. One measure applied is the coefficient of variation CV, which is defined by the ratio of the standard deviation divided by the expectation. The following errors are now defined:

$$\varepsilon = \eta_m - \eta_c \quad [7a]$$

$$\varepsilon_m = \eta_m - \eta_t \quad [7b]$$

$$\varepsilon_m = \eta_m - \eta_t \quad [7c]$$

$$\varepsilon_c = \eta_c - \eta_e \quad [7d]$$

Here η_m is the measured viscosity, η_t the (unknown) true one and η_c the viscosity calculated e.g. by one of the approaches shown in this paper. Further η_e denotes the viscosity calculated by an equation which results in perfect uncorrelated residue with zero expectation. Fig. 7 and 8 show that this is to some degree fulfilled by equ. (6). The coefficient of variance of the total residue ε is shown in Tab. 3. The measurement error ε_m consists of the error due to the viscometric measurement and that caused by the variance of the measured ribbon length. Both have been estimated for the measurements of this investigation, based on three repetitions of each viscometric measurement, and five repetitions of each IPT test. This results in a total coefficient of variation $CV(\varepsilon_m) = 0.123$. Further

for the total error CV(ε) = 0.226 was received. The deviation of these two values is contributed by the variances of the systematic errors ε_e and error ε_c. They amount to a further coefficient of variation of 0.190, which exceeds that of the measurement. What does this mean for the expectation E(ε) of the total error ε of an IPT test performed by several repetitions? In the best case (no systematic error of viscosity measurement and uncorrelated residues of the evaluation formula of IPT with zero expectation) $E(\varepsilon_m)$ and $E(\varepsilon_c)$ will tend to vanish with increasing number of repetitions. This will unfortunately not hold for ε_e. It will not depend on the number of repetitions and cannot further be decreased with the formulas quoted here. The case $\varepsilon_e \neq 0$ means the true viscosity relation has not been found, even though the residue distribution is satisfying. It is assumed that ε_e is caused by further parameters impacting the ribbon length and acting additionally to a single viscosity at a given temperature. On the one hand this may be the viscosity change with temperature (related to the activation energy of viscous flow) as not only a single viscosity at one temperature influences. Further also partial crystallization may have an impact.

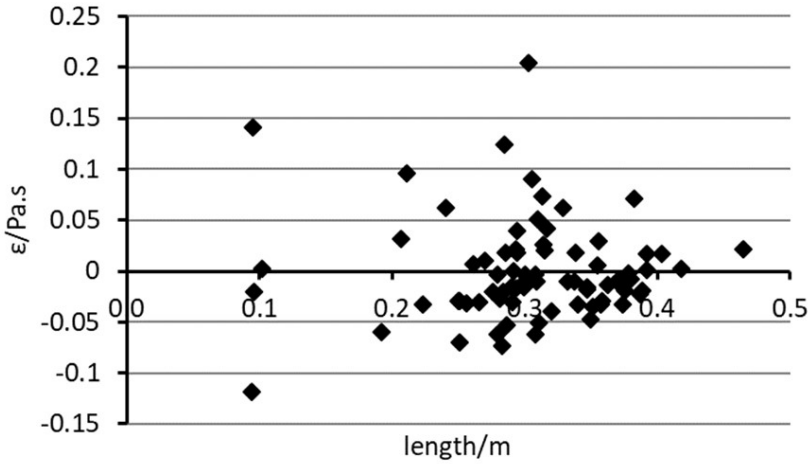

Fig.7 - Residues of the hyperbolic approach.

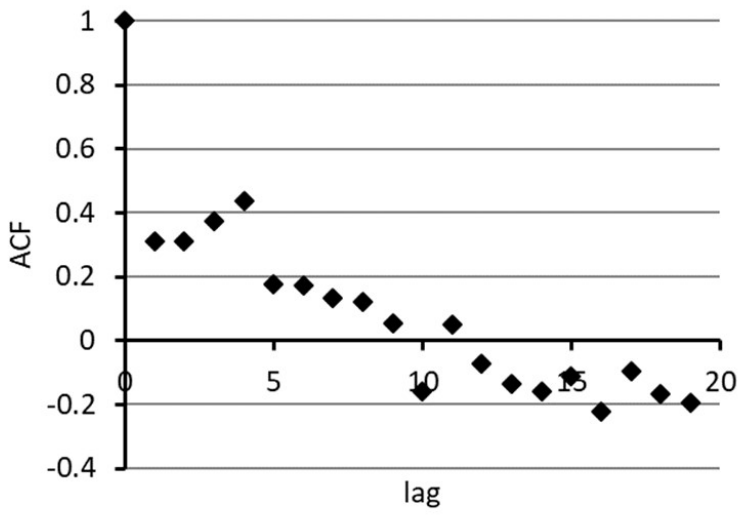

Fig.8 - Autocorrelation function of the residues.

CONCLUSION

The formulas according to Dey (n=-1) and Bazan (n=3) are both not generally valid models, viz they cannot be applied for arbitrary slags. The equation of Dey is close to the optimum fit for the individual data set of the author. Available modelling equations are well able to reproduce data without systematic error (satisfying distribution of residues). In this study, the coefficient of determination B is above 97% for all investigated approaches and data sets. Nevertheless, a not revealed error due to not considered variables remains. It may be relatively large, its coefficient of variation amount to 0.19 for the investigation and would only allow sufficient accuracy for relatively high viscosities. The coefficient of variation caused by the unexplained error would be below 10% in the range 0.499 to 1.736 Pa.s and below 5 % in the region 0.9888 to 1.736 Pa.s. To this, the stochastic measurement error caused by the variation of the ribbon length adds. More accurate data of Dey et al may be related to the application of calculated instead of measured viscosities. The unexplained error is expected to be due to the viscosity change caused by cooling during ribbon formation and partly due to crystallisation. Restriction of the whole collective to groups with respect to mould powders types, suppliers, similar compositions or viscosity groups may be suitable to reduce the unexplained variance.

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