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INFLUENCE OF SPECIFIC SURFACE OF LIGNITE FLUIDAL ASHES ON RHEOLOGICAL PROPERTIES OF SEALING SLURRIES

WPŁYW POWIERZCHNI WŁAŚCIWEJ POPIOŁÓW FLUIDALNYCH Z WĘGLA BRUNATNEGO NA WŁAŚCIWOŚCI REOLOGICZNE ZACZYNÓW USZCZELNIAJĄCYCH

New generation fly ashes come from the combustion of coal in fluid-bed furnaces with simultaneous sulphur-removal from gases at ca. 850°C. Accordingly, all produced ashes basically differ in their physicochemical properties from the traditional silica ones. The aim of the laboratory analyses was determining the influence of specific surface and granular composition of fluidal ash on rheological properties of slurries used for sealing up the ground and rock mass media with hole injection methods, geoengineering works and cementing casing pipes in deep boreholes.

Fluidal ash from the combustion of lignite contain active Puzzolan appearing in the form of dehydrated clayey minerals and active components activating the process of hydration ashes, i.e. CaO, anhydrite II and $CaCO_3$. The ashes have a weak point, i.e. their high water diment, which the desired rheological properties related with the range of their propagation in the rock mass cannot not be acquired for injection works in the traditional sealing slurries technology. Increasing the water-to-mixture ratio should eliminate this feature of fluidal ashes.

Laboratory analyses were performed for slurries based on metallurgical cement CEM III/A 32,5 having water-to-mixture ratios: 0.5; 0.6 ; 0.7 and 0.8; the fluidal ash concentration in the slurries was 30 wt.% (with respect to the mass of dry cement).

Basing on the obtained results there were determined optimum recipes of sealing slurries in view of their rheological parameters which could be applied both in drilling technologies (cementing casing pipes, closing of boreholes, plugging) and in geoengineering works related with sealing up and reinforcing ground and rock mass media.

Keywords: Cementing Wells, Rheological Properties, Fluidal Ash

Popioły lotne nowej generacji powstają ze spalania węgla w kotłach fluidalnych z równoczesnym odsiarczaniem gazów. Proces ten przebiega w temperaturze około 850°C. Zatem powstałe popioły różnią się swoimi właściwościami fizykochemicznymi w sposób zasadniczy od tradycyjnych popiołów krzemionkowych. Celem przeprowadzonych badań laboratoryjnych było określenie wpływu powierzchni

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właściwej oraz składu ziarnowego popiołów fluidalnych na właściwości reologiczne zaczynów służących do uszczelniania ośrodka gruntowego oraz masywu skalnego metodami iniekcji otworowej, do prac geoinżynieryjnych oraz cementowania kolumn rur okładzinowych w głębokich otworach wiertniczych.

Popioły fluidalne powstałe ze spalania węgla brunatnego zawierają w swoim składzie aktywną pucolanę występującą w formie zdehydratyzowanych minerałów ilastych oraz aktywne składniki aktywujące proces hydratacji tych popiołów jakimi są CaO, anhydryt II oraz $CaCO_3$. Słabą stroną tych popiołów jest ich duża wodożądność a tym samym w tradycyjnej technologii zaczynów uszczelniających do prac iniekcyjnych nie uzyskano by pożądanych właściwości reologicznych związanych z promieniem ich rozpływu w górotworze. Zwiększenie współczynnika wodno-mieszaninowego, powinno wyeliminować tą słabszą właściwość popiołów fluidalnych.

Badania laboratoryjne przeprowadzono na zaczynach sporządzonych na osnowie cementu hutniczego CEM III/A 32,5 o współczynnikach wodno-mieszaninowych 0.5; 0.6; 0.7 i 0.8, zaś koncentracja popiołu fluidalnego w zaczynach wynosiła 30% (wagowo w stosunku do masy suchego cementu).

Na podstawie uzyskanych wyników z badań określono optymalne receptury zaczynów uszczelniających ze względu na ich właściwości reologiczne, które mogą być zastosowane zarówno w technologiach wiertniczych (cementowanie kolumn rur, likwidacja odwiertów, wykonywanie korków) jak i w pracach geoinżynieryjnych związanych z uszczelnieniem i wzmocnieniem ośrodka gruntowego lub masywu skalnego.

Słowa kluczowe: cementowanie otworów, właściwości reologiczne, popioły fluidalne

1. Introduction

Rheological properties of sealing slurries are very important for designing and performing sealing and reinforcement works in ground and rock mass medium with the use of drilling technologies. High efficiency of sealing columns of casing pipes in deep boreholes and also sealing of the rock mass with borehole injection methods can be provided if rheological parameters of sealing slurries are selected properly in view of (Stryczek, 2009; Wiśniowski, 2001):

- Formation conditions of sealed ground and rock mass,
- Geometry of borehole and circulation system,
- Interrelations between volume of injected slurry and thus created pressure loss, especially in the sealed medium.

The use of sealing slurries based on Portland cement frequently turns out disadvantageous. They have a number of shortcomings, e.g. long time of binding and inappropriate rheological parameters. Unfavorable properties of cement slurries can be significantly improved by introducing ground slag granulate and other pre-selected mineral additives. Henceforth, investigations have been recently carried out to develop binders and slurries for special new-generation binders. This type of slurries is exclusively based on inorganic components.

Mineral additives introduced to cement may result in a modification of a number of properties: longer time of binding, lower kinetics of heat production and dynamics of early strength growth, higher resistivity to corrosive environment (Jasiczak, 2003; Kon 2000; Pinka, 2006).

One of such solutions is adding fluidal ash, which significantly lowers the cost of reinforcing and sealing operations. The addition of fluidal ash to cement lowers the cost of the slurry by 30÷40%, depending on the cement.

The strict UE directives on environmental protection and the increasing cost of electric-plant waste deposition urge the producers of coal-based energy to search for a new rational and economic way of disposing of the waste. One of the products of coal combustion is ash, which may be successfully used as a sealing mixtures component.

2. Fluidal ash

Fluidization process lies in suspending solid particles present in the upstreaming fluid. The deposit of solid particles is intensely mixed up, assuming the semi-suspended (fluidal) form, and revealing a number of features typical of fluids. In this state the contact surface between grains and fluid is very developed, thanks to which thermal and diffusion processes between solid and liquid or gaseous are facilitated. Using the above grain sizes and specific speed of gas, high solids concentrations in the solid-gas system can be obtained. In such conditions the solid body is intensely mixed up, which together with strongly developed surface of the body create perfect conditions for penetration of heat and movement of mass.

In the fluid bed burner the lower quality, high-sulphur fuels can be combusted, with simultaneous lower sulphur oxides and nitrogen emissions to atmosphere in the course of combustion. During combustion in the fluid-bed burner coal is mixed up with inert material, therefore a low combustion temperature of about 850°C can be obtained, i.e. below the temperature of softening for ash. This results in lowering the amount of contaminations on the heating surfaces, which has great influence on the magnitude of NO_x emissions (the lower the temperature, the lower is the temperature of nitrogen oxides (NO_x) formation). The dolomite or limestone admixed to fuel bind SO_2 produced in the course of sulphur burning in the bed. Owing to this, the sulphur oxide emission can be reduced six times at the burner level. As the limestone has its optimum binding of SO_2 within the temperature range of 800-900°C, its use is much lower than when applying other methods of sulphur removal from discharge gases beyond the boiler. Another advantage of fluid-bed furnace is intense exchange of heat, which facilitates decreasing of the heating surface in the boiler. Fluid-bed burners may be used both in newly built furnaces and the modernized ones (Brylicki, 2001; Kurdowski, 1991).

The main premises determining the high attractiveness of ground fluidal waste for construction industry is mainly its high Puzzolan activity and good grindability. The high Puzzolan activity of fluidal ash stems from the dominating role of amorphous and weakly crystallized products of clayey minerals dehydration, i.e. illite, montmorillonite, kaolinite or chlorite. The Puzzolan activity of fluidal ash depends on the content of enclosed reactive components (SiO_2, Al_2O_3) as compared to the calcium hydrate produced in the course of cement hydration. The free calcium, being one of the elements of fluidal ash, accelerates the time of cement binding, though cement should not contain excessive amounts of free CaO. The high grindability of cements with fluidal ash enables obtaining a very developed specific surface, which makes grinding less energy-consuming, and thus more economic. The big specific surface of cement slurries with fluidal ash intensifies the course of chemical reactions even in the initial stages of cement hydration, which results in good dynamics of cement strength growth from the first days of its setting. By introducing fluidal ash to sealing slurry on behalf of cement lowers its unit price by 30÷40% with respect to classic cement slurries (Małolepszy, 2010; Śliwiński, 1999).

Owing to the fact that fluidal ash is a waste material, its use in the production of special-purpose binders requires constant and detailed supervision of its physicochemical properties.

3. Laboratory analyses

Laboratory analyses of rheological parameters of sealing slurries were prepared on the basis of the following standards:

1. PN-EN 197-1: 2002, Cement. Part 1. Composition, requirements and congruence criteria for common use cements.
2. PN-EN ISO 10426-1. Oil and gas industry. Cements and materials for cementing boreholes. Part 1. Specification. 2006
3. PN-EN ISO 10426-2. Oil and gas industry. Cements and materials for cementing boreholes. Part 2: Investigation of drilling cements. 2006.

The laboratory tests were oriented to checking out the usability of metallurgical cement CEM III/A 32,5 and fly ash from fluidal combustion of lignite as a mineral additive of slurries used for sealing and reinforcing rock mass (Górażdże Cement, 2002). The following variables were taken into account in the analyses:

a) water-to-cement ratio,
b) specific surface of fluidal ash.

The water-to-mixture (cement with ash) ratio for analyzed sealing slurries was the following: 0.5; 0.6; 0.7 and 0.8. Fluidal ash was added to Portland cement in 30 wt.% with respect to cement dry mass.

The following kinds of fly ashes from fluidal combustion of lignite have been used for laboratory analyses (Brylicki, 2001; Małolepszy, 2010):

A – fluidal ash (averaged fly ash being an end by-product of waste gases de-dusting process),
B – fluidal ash – de-dusting zone I,
C – fluidal ash – de-dusting zone II,
D – fluidal ash – de-dusting zone III.

The density of ash established with the pycnometric method and oil was equal to:

Type of ash	A	B	C	D
Density [g/dm^3]	2.66	2.65	2.67	2.65

The specific surface determined on the basis of isotherm BET and with the Blaine method was equal to:

Type of ash	A	B	C	D
Specific surface BET [cm^2/g]	41500	50300	62100	74200
Specific surface Blaine[cm^2/g]	7100	3540	11950	15470

whereas the grain size distribution was established with the use of a laser particle-size analyzer Malvern Mastersizer 2000 (Fig. 1).

Rheological properties (plastic viscosity, apparent viscosity, yield point) of the analyzed sealing slurries were established with a rotary viscometer having coaxial cylinders of type Chan – 35 API Viscometer – Tulsa, Oklahoma USA EG.G Chandler Engineering with twelve rotational speeds (600, 300, 200, 100, 60, 30, 20, 10, 6, 3, 2, 1 rpm, which corresponds to the following shear rates: 1022.04; 511.02; 340.7; 170.4; 102.2; 51.1; 34.08; 17.04; 10.22; 5.11; 3.41; 1.70 s^{-1}).

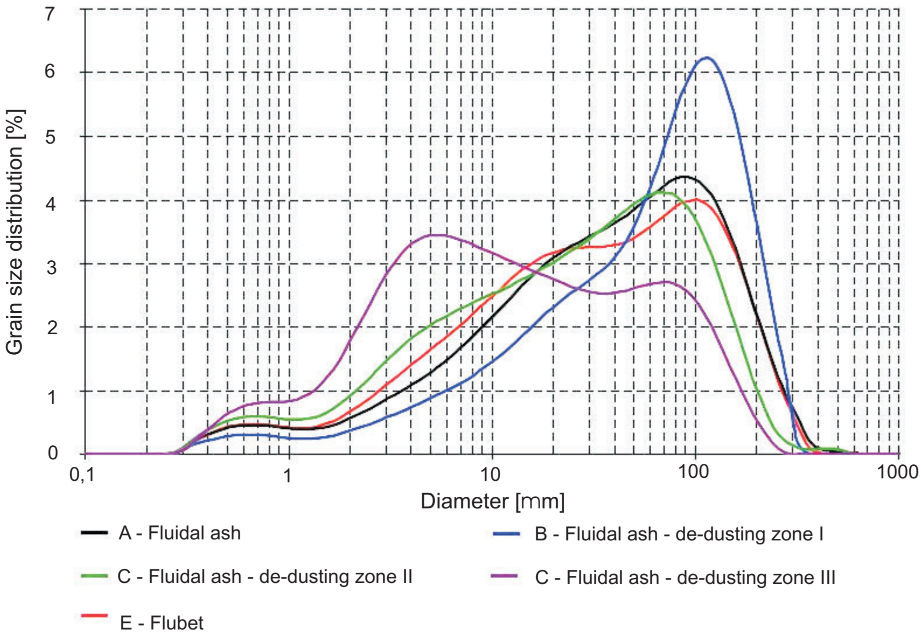

Fig. 1. Distribution of grain size of fly ash used in analyses – differential curves (Małolepszy, 2010)

The selection of proper rheological model of analyzed sealing slurries with admixed fly ash from lignite combustion lied in determining a rheological curve thanks to which the results of measurements in the coordinates system: shear rate (γ) – shear stress (τ) could be described better.

Rheological parameters for particular models were determined using the regression analysis method. Then statistical tests were used for determining the optimum rheological model for a given recipe of sealing slurry (Wiśniowski, 2006; Wiśniowski, 2007).

To facilitate calculations related with establishing optimum rheological models for analyzed slurries, a computer program "Rheosolution" has been used, a modified version of „Flow – Fluid Coef" (Wiśniowski, 2001; Wiśniowski, 2006). This software is owned by the Department of Drilling and Geoengineering, Faculty of Drilling, Oil and Gas AGH-UST and is used for scientific and research purposes.

4. Results of laboratory analyses

Parameters and rheological models of slurry solely based on metallurgical cement CEM III/A 32,5 for various water-to-cement ratios have been presented in table 1.

Rheological properties of slurries with 30% admixture of fly ash from fluidal combustion of lignite type A (averaged ash) have been presented in table 2.

The results of analyses of rheological parameters of cement slurries with admixture of 30% fly ash of type B, C and D have been listed in tables 3, 4 and 5.

TABLE 1

Rheological parameters of cement slurries having various water-to-cement ratios determined at temp. 20°C for various rheological models

Rheological parameters \ Water-to-cement		0.5	0.6	0.7	0.8
Newton's Model	Newtonian absolute viscosity [Pa · s]	0.901	0.056	0.038	0.023
	Correlation coefficient [-]	0.857	0.885	0.918	0.823
Bingham's Model	Plastic viscosity [Pa · s]	0.071	0.045	0.031	0.018
	Yield point [Pa]	12.211	7.047	4.279	3.512
	Correlation coefficient [-]	0.984	0.992	0.997	0.995
Ostwald de Waele's Model	Consistency coefficient [Pa · s^n]	4.459	2.946	1.969	1.957
	Exponent [-]	0.388	0.366	0.359	0.283
	Correlation coefficient [-]	0.972	0.946	0.917	0.880
Casson's Model	Casson's viscosity [Pa · s]	0.041	0.025	0.018	0.008
	Yield point [Pa]	6.971	4.183	2.558	2.469
	Correlation coefficient [-]	0.996	0.998	0.998	0.986
Herschel-Bulkley's Model	Yield point[Pa]	7.554	5.347	3.591	3.544
	Consistency coefficient [Pa · s^n]	0.662	0.205	0.083	0.016
	Exponent [-]	0.677	0.779	0.858	1.014
	Correlation coefficient [-]	0.996	0.997	0.999	0.995
Apparent viscosity at 1022.04 [s^{-1}] [Pa · s]		0.080	0.050	0.035	0.022

TABLE 2

Rheological parameters of cement slurries (CEM III/A 32,5 + 30% ash A) having different water-to-cement ratios determined at temp. 20°C for various rheological models

Rheological parameters \ Water-to-cement		0.5	0.6	0.7	0.8
Newton's Model	Newtonian absolute viscosity [Pa · s]	0.307	0.111	0.05	0.029
	Correlation coefficient [-]	0.971	0.965	0.953	0.925
Bingham's Model	Plastic viscosity [Pa · s]	0.273	0.098	0.047	0.024
	Yield point [Pa]	7.398	8.275	4.877	3.134
	Correlation coefficient [-]	0.994	0.994	0.997	0.999
Ostwald de Waele's Model	Consistency coefficient [Pa · s^n]	2.351	2.455	1.992	1.760
	Exponent [-]	0.616	0.504	0.415	0.317
	Correlation coefficient [-]	0.991	0.963	0.925	0.856
Casson's Model	Casson's viscosity [Pa · s]	0.210	0.068	0.029	0.012
	Yield point [Pa]	2.412	3.668	2.654	2.074
	Correlation coefficient [-]	0.999	0.998	0.997	0.988
Herschel-Bulkley's Model	Yield point[Pa]	2.826	5.806	4.023	3.265
	Consistency coefficient [Pa · s^n]	1.041	0.294	0.091	0.017
	Exponent [-]	0.771	0.841	0.906	1.045
	Correlation coefficient [-]	0.999	0.997	0.998	0.999
Apparent viscosity at 1022.04 [s^{-1}] [Pa · s]		no	0.105	0.052	0.027

TABLE 3

Rheological parameters of cement slurries (CEM III/A 32,5 + 30% ash type B)
of various water-to-cement ratio determined at temp. 20°C for various rheological models

Rheological parameters \ Water-to-cement		0.5	0.6	0.7	0.8
Newton's Model	Newtonian absolute viscosity [Pa · s]	0.300	0.237	0.120	0.054
	Correlation coefficient [-]	0.943	0.944	0.936	0.929
Bingham's Model	Plastic viscosity [Pa · s]	0.258	0.203	0.103	0.045
	Yield point [Pa]	9.211	11.297	10.945	5.627
	Correlation coefficient [-]	0.983	0.985	0.983	0.995
Ostwald de Waele's Model	Consistency coefficient [Pa · s^n]	2.669	3.018	2.440	2.193
	Exponent [-]	0.596	0.564	0.536	0.405
	Correlation coefficient [-]	0.998	0.998	0.996	0.943
Casson's Model	Casson's viscosity [Pa · s]	0.196	0.150	0.075	0.027
	Yield point [Pa]	3.069	4.199	4.351	3.125
	Correlation coefficient [-]	0.992	0.994	0.994	0.998
Herschel-Bulkley's Model	Yield point[Pa]	0.540	1.509	3.153	4.486
	Consistency coefficient [Pa · s^n]	2.220	2.152	1.183	0.136
	Exponent [-]	0.636	0.625	0.650	0.840
	Correlation coefficient [-]	0.999	1	0.999	0.997
Apparent viscosity at 1022.04 [s^{-1}] [Pa · s]		no	no	0.106	0.050

TABLE 4

Rheological parameters of cement slurries (CEM III/A 32,5 + 30% ash type C)
of various water-to-cement ratio determined at temp. 20°C for various rheological models

Rheological parameters \ Water-to-cement		0.5	0.6	0.7	0.8
Newton's Model	Newtonian absolute viscosity [Pa · s]	0.817	1.444	0.082	0.043
	Correlation coefficient [-]	0.942	0.880	0.849	0.846
Bingham's Model	Plastic viscosity [Pa · s]	0.681	0.118	0.065	0.034
	Yield point [Pa]	15.026	17.208	10.976	5.971
	Correlation coefficient [-]	0.993	0.969	0.968	0.984
Ostwald de Waele's Model	Consistency coefficient [Pa · s^n]	6.878	4.534	3.050	2.369
	Exponent [-]	0.541	0.469	0.446	0.366
	Correlation coefficient [-]	0.989	0.996	0.997	0.966
Casson's Model	Casson's viscosity [Pa · s]	0.463	0.081	0.042	0.019
	Yield point [Pa]	6.186	7.872	5.443	3.488
	Correlation coefficient [-]	0.999	0.987	0.987	0.998
Herschel-Bulkley's Model	Yield point[Pa]	6.480	3.269	2.751	3.588
	Consistency coefficient [Pa · s^n]	2.833	2.738	1.691	0.350
	Exponent [-]	0.728	0.548	0.533	0.661
	Correlation coefficient [-]	0.999	0.999	0.999	0.999
Apparent viscosity at 1022.04 [s^{-1}] [Pa · s]		no	0.122	0.070	0.037

TABLE 5

Rheological parameters of cement slurries (CEM III/A 32,5 + 30% ash type D) of various water-to-cement ratio determined at temp. 20°C for various rheological models (fig. 2)

Rheological parameters	Water-to-cement	0.5	0.6	0.7	0.8
Newton's Model	Newtonian absolute viscosity [Pa · s]	0.614	0.321	0.108	0.023
	Correlation coefficient [-]	0.950	0.979	0.896	0.924
Bingham's Model	Plastic viscosity [Pa · s]	0.519	0.292	0.091	0.019
	Yield point [Pa]	10.559	9.514	11.157	2.365
	Correlation coefficient [-]	0.994	0.993	0.957	0.989
Ostwald de Waele's Model	Consistency coefficient [Pa · s^n]	4.624	2.319	2.104	1.111
	Exponent [-]	0.567	0.655	0.551	0.364
	Correlation coefficient [-]	0.992	0.995	0.998	0.891
Casson's Model	Casson's viscosity [Pa · s]	0.364	0.239	0.071	0.010
	Yield point [Pa]	4.098	2.596	3.963	1.461
	Correlation coefficient [-]	0.999	0.997	0.972	0.983
Herschel-Bulkley's Model	Yield point[Pa]	4.326	1.487	-1.908	2.402
	Consistency coefficient [Pa · s^n]	2.067	1.497	2.892	0.017
	Exponent [-]	0.736	0.739	0.505	1.016
	Correlation coefficient [-]	0.999	0.999	0.998	0.989
Apparent viscosity at 1022.04 [s^{-1}] [Pa · s]		no	no	0.089	0.021

5. Closing conclusions

The laboratory tests and analyses of the obtained results reveal that 30 wt.% admixture of fly ash from fluidal combustion of lignite to sealing slurry based on metallurgical cement CEM III/A 32,5 deteriorates rheological parameters of the slurry for each of the analyzed water-to-mixture coefficients.

Owing to their parameters and rheological properties, the analyzed sealing slurries in 80% of cases can be best described with the Herschel-Bulkley's rheological model.

The linear models (Newton's and Bingham's) of the analyzed sealing slurries should not be used for precise calculation of pressure loss which may occur in the process of sealing up the casing pipes, especially in deep boreholes and when designing the range of propagation of the slurry in the ground when performing injection operations.

Among the analyzed four types of fluidal fly ashes the slurries based on ash type A had the biggest fluidity, and slurries based on ash type B (zone I) had the lowest fluidity for the assumed water-to-mixture ratios (30 wt.%).

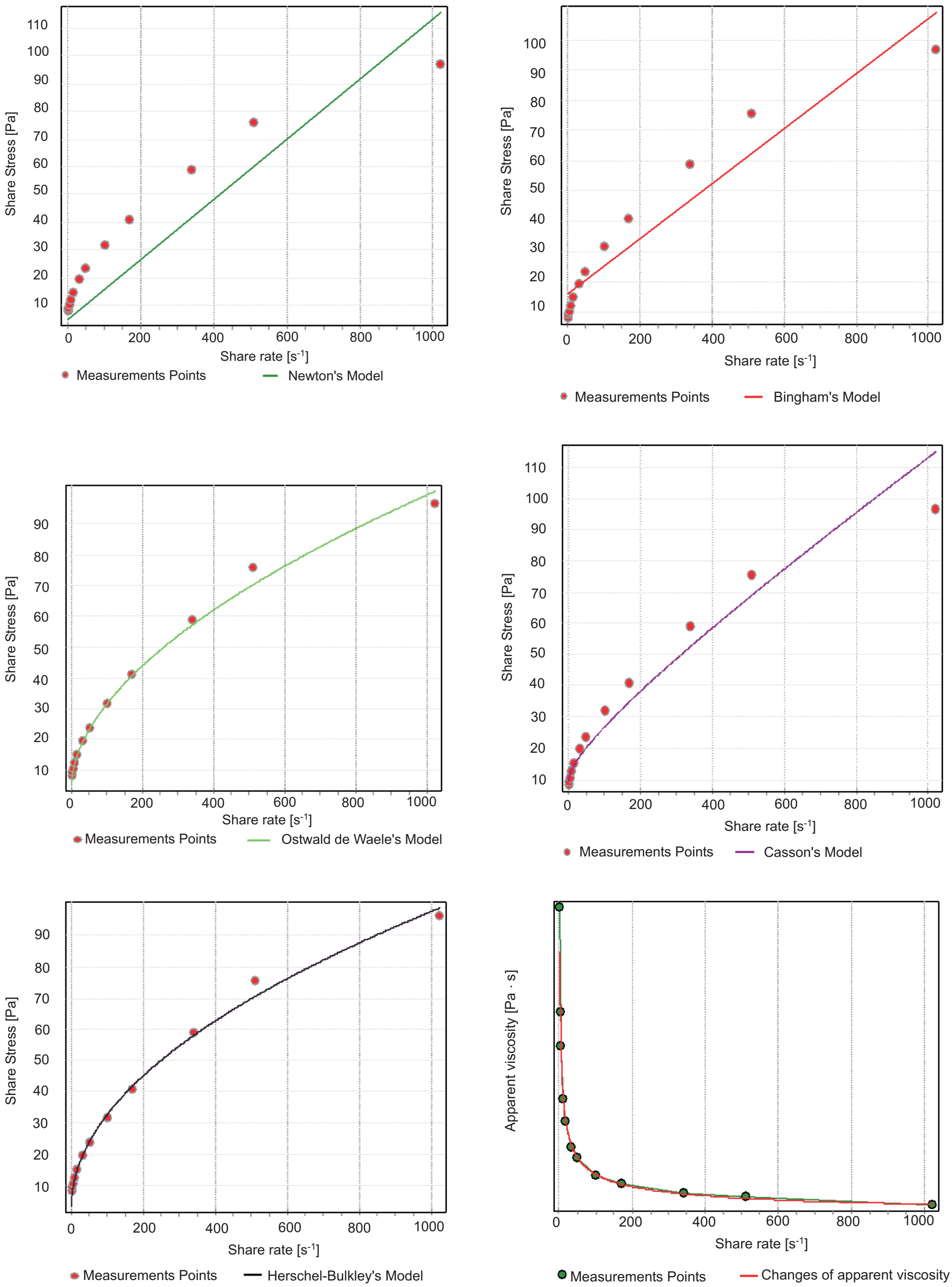

Fig. 2. Exemplary plots of tangential stress vs. shear rate and changes of apparent viscosity for metallurgical cement CEM III/A 32,5 with 30% fly ash type D for water-to-cement ratio = 0.7

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