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RHEOLOGICAL PROPERTIES OF COAL WATER SLURRIES CONTAINING PETROCHEMICALS

by

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In this paper, the rheological properties (viscosity, shear stress, and lamination) of the promising coal-water slurries containing petrochemicals based on filter cakes have been investigated experimentally. The latter are a mixture of water and solid organic wastes produced during coal preparation. It was established that the rheological properties of coal-water slurries containing petrochemicals depend on concentrations and properties of the main components (liquid and solid, flammable, and non-flammable), wetting agents (plasticizers), as well as the storage time and conditions. The rheological properties of the fuel slurries under study were compared with the known properties of coal-water slurries based on different ranks of coal and found to match or even surpass them. Coal processing wastes have high potential as prospective components of coal-water slurries containing petrochemicals in terms of not only their environmental, economic and energy performance but also their rheological properties.

Key words: *coal-water slurry containing petrochemicals, filter cakes, viscosity, waste flammable liquids, rheological properties, lamination*

Introduction

Although the global scientific community is working hard to develop and implement energy-efficient technologies, the consumption of energy resources is growing non-linearly each year together with the increasing needs of productions [1-3]. As a result, the total amount of mineral resources (coal, gas, and oil) being extracted is rapidly growing as well [1-4]. Over the recent years, the oil prices and production have been extremely unstable due to a number of well-known factors. In this situation, coal may become the main energy source for the coming decades. One of the reasons for these forecasts [1, 2] lies in the annual reports of the declining amount of oil and gas in the earth's crust.

Coal fuels are notorious for their drawbacks [5-8] as compared to oil and gas. First, coal combustion produces maximum anthropogenic emissions and ash residue. Second, coal processing generates wastes in large quantities and their usage is limited for a number of technological, economic, and environmental reasons. Third, coal transportation is only possible by rail, water, and road, which is much more expensive than using pipe-line transport. Therefore, it seems sensible to develop ways to minimize these drawbacks. Among the most promising technologies in this respect are the approaches of using coal-water slurries (CWS) [9-11] and coal-water slurries containing petrochemical (CWSP) [12-15]. These fuels can be

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transported by pipe-lines. They show high fire and explosion safety and produce anthropogenic emissions in lower concentrations due to water in their composition. Unlike with coal dust, there is no chance of unregulated fires at the fuel preparation and transportation stages, since this fuel contains a considerable proportion of water, which has high vaporization heat. The components of such fuels can be various coal processing wastes or low-rank coals. Other prospective components of CWS and CWSP include [12-15] waste combustible liquids, oil sludge, resins *, etc.*

Oil and coal processing as well as transportation and storage result in numerous wastes: oil sludge and filter cakes [2-4]. The volume of these wastes ranges from 8% to 15% of the total energy production, depending on deposits, processing technologies, and production rate [16-18]. For example, China produced 1.84 billionns of coal and lignite and around 162 millionns of cakes in 2015. These proportions are even more impressive for Russia and the USA. The said issues are of particular interest in Russia, where most of these wastes are stored outdoors [16-18].

Filter cakes and oil sludge pose a serious danger to the environment [19]. Some of these wastes are disposed of but the bulk is not recycled at all. The world has already accumulated more than 1 billionns of sludge. Moreover, various industries annually produce tens of millions of tons of liquid combustible wastes (*e. g.*, used motor, turbine, transformer, compressor oils *, etc.*) [19].

Filter cakes are promising, though understudied, fuel components in terms of economic, environmental and energy performance indicators [12-15] as well as fire and explosion safety. The main information gap concerns the rheological properties of CWSP fuels based on these filter cakes.

Rheological properties of various fluids play very important practical application [20-22] for its storage, transportation and atomization, because of the purpose of this work is to accumulate the experimental database on the rheological properties of CWSP fuels based on typical coal processing wastes (filter cakes).

Fuel components

Production of filter cakes during coal processing

The key components of the CWSP under study were filter cakes (cakes) – coal processing wastes from coal preparation plants in Kemerovo Region, Russia. Filter cakes are the result of coal processing, when coal slurry thickens with the help of surfactants (flocculants) in circular thickeners. The slurry with high solids concentration is then filtered and excess moisture is removed in the process. A moist high-ash remainder is called a filter cake.

Typical filter cakes of flame, gas, low-caking, non-banking, and coking coals were considered. These coals are widely used in by-product coke industry. The solid particles of cakes in a CWSP were 80-120 μm in size [12-15].

The production of filter cakes at coal processing plants is complicated and comprises many stages. In particular, the main features can be outlined. Coal preparation is a sequence of operations involved in coal processing and aimed at separating it from impurities that prevent from its further use. Impurities are mostly mineral components and sulfur-containing substances. The coal preparation produces a concentrate, which has the highest content of pure coal. Industrial product, which contains bone and rock which has the highest content of mineral impurities.

Key methods of coal preparation: gravity separation based on the differences in the component density, flotation based on different natural or chemical wettability of components,

magnetic separation based on different magnetic susceptibility of components, electric separation based on different electric properties of components, and special types, which are rarely used with coals (x-ray separation, processing according to shape and friction, magnetohydrodynamic processing, chemical processing, *etc.*). A rational choice in favor of one or several of these methods is primarily based on the properties of coal, methods and technologies of preparation, as well as economic performance indicators. Currently, the most widespread methods in the world are gravity separation and flotation.

Flotation made it possible to increase the yield of coking concentrate and reduce its ash content. Flotation is classified into oil, film and froth one. Oil and film flotation is not popular for coal processing due to its insufficient effectiveness and low machine capacity. Froth flotation has won general recognition and industrial prevalence. In an aerated pulp, air bubbles rise up and some relatively clean coal particles become bound to these bubbles. The bubbles reach the surface with the coal particles. The resulting flotation froth is then collected from the surface using special devices. Wet particles are not attached to air bubbles and remain in the pulp. To intensify froth flotation, special reagents are added to the pulp. Froth flotation is used to separate sludge particles sized under 0.5 mm. There are hydrophilic (wettable) and hydrophobic (non-wettable) particles. Coal particles are among hydrophobic ones. It should be mentioned that different coal types have different hydrophobic characteristics. For example, brown coals are wettable; therefore, they are impossible to process by flotation. Flotation reagents can be of three types: apolar, heteropolar, and dual action reagents.

The CWS and CWSP fuels are currently prepared from the wastes produced on a belt filter press. Filter cakes produced there is a waste that is usually dumped. The ash and moisture content of those cakes may vary in a very wide range depending on many factors. The current annual production of coal in Kemerovo Region is about 215 millionns (as of 2015). On average, about 60-70% of coal is cleaned (for coking coal, this percentage reaches about 90-95%). There are plans to drive these values to 85% by 2020 with the coal production of 230 millionns a year. The relative rate of flotation wastes is 120-130 kg per tonnes of run-of-mine coal and that of overburden, 410 kg per tonnes of run-of-mine coal (the data is tentative). Consequently, the annual production of filter cakes will multiply and the problem of their utilization will become ever more urgent. These issues are not only typical of Russia but also of China, India, Japan and other countries.

Production of filter cakes during coal processing

Solid particles of filter cakes in the CWSP were 80-120 μm in size. See tab. 1 for the results obtained from the proximate analysis of the filter cakes under study, tab. 2 for the ultimate analysis, and tab. 3 for ash content. Table 4 presents the content of dry substance in filter cakes when they are produced at preparation plants.

Table 1. Results of proximate analysis of filter cakes

Sample	W^a [%]	A^d [%]	V^{daf} [%]	$Q^d_{s,V}$ [MJ/kg]
Filter cake of flame coal	56.8	36.99	41.47	19.26
Filter cake of gas coal	53.0	33.82	43.11	22.17
Filter cake of coking coal	43.5	26.46	23.08	24.83
Filter cake of low-caking coal	37.9	50.89	30.16	15.23
Filter cake of non-baking coal	39.1	21.20	16.09	26.92

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Table 2. Results of ultimate analysis of filter cakes

Sample	C^{daf} [%]	H^{daf} [%]	N^{daf} [%]	S^{daf} [%]	O^{daf} [%]
Filter cake of flame coal	73.27	4.90	0.02	0.22	21.59
Filter cake of gas coal	75.12	4.64	0.02	0.23	19.99
Filter cake of coking coal	79.79	4.49	1.84	0.87	12.70
Filter cake of low-caking coal	77.30	4.78	1.93	0.33	15.32
Filter cake of non-baking coal	87.97	4.10	2.23	0.53	5.03

Table 3. Results of ultimate analysis of the ash in filter cakes

Component	Mass fraction of component [%]				
	Filter cake of flame coal	Filter cake of gas coal	Filter cake of coking coal	Filter cake of low-caking coal	Filter cake of non-baking coal
Na_2O	2.11	2.07	1.76	0.74	0.76
MgO	2.13	2.17	3.03	1.95	1.85
Al_2O_3	22.46	22.11	19.76	25.14	27.25
SiO_2	61.43	57.70	57.11	55.74	50.09
P_2O_5	0.49	0.54	0.25	0.19	1.30
SO_3	0.89	1.56	2.75	1.61	2.39
K_2O	3.82	4.16	4.09	4.26	2.67
CaO	2.23	4.01	3.03	3.70	3.61
TiO_2	0.99	1.14	1.10	0.98	1.50
Fe_2O_3	3.40	4.49	7.08	5.64	8.53

Table 4. Main properties of filter cakes (in jelly state) as received during coal preparation

Filter cake at its initial (wet) state	Mass fraction of dry substance [%]	$Q^a_{s,V}$ [MJkg^{-1}]
Filter cake of coking coal	56.5	14.1
Filter cake of flame coal	43.2	8.3
Filter cake of gas coal	46.9	10.4
Filter cake of low-caking coal	62.1	9.5
Filter cake of non-baking coal	60.9	16.4

Properties of liquid fuel components of CWSP

    Two most widespread liquid fuel components were used to prepare CWSP based on filter cakes: fuel oil and used turbine oil. Table 5 gives the main characteristics of these components, which significantly affect CWSP properties [12-15].

Table 5. Results obtained from the analysis of liquid fuel components

Component	ρ [kgm^{-3}] at 293 K	W^a [%]	A^d [%]	T_f [K]	T_{ign} [K]	$Q^a_{s,V}$, [MJkg^{-1}]
Used turbine oil	868	–	0.03	448	466	45.1
Fuel oil	1000	6.12	4.06	438	513	39.4

The authors of [12-15] show the differences in the ignition characteristics of CWSP fuels based on widespread waste industrial oils – motor, turbine, transformer, compressor oils, *etc.*, heavy coal-tar products, resins, oil sludge, oils, oil-water emulsions, as well as a widely spread additive to CWS: fuel oil. It follows from the research findings [12-15] that turbine oil and fuel oil used in this work have mediocre ignition characteristics as compared to all the promising liquid fuel components of CWSP fuels.

Preparation of CWSP fuels

There are several methods for CWS and CWSP preparation [23]: using homogenizers or dispersers and using ball mills or cavitators. The preparation of slurry fuels using a homogenizer is usually conducted in the following way. According to their varied relative mass concentration, CWSP components are loaded into the homogenizer tank for mixing, which lasts for up to 10 minutes. The second way of CWSP preparation is based on coal cavitation in a water medium, molecular destruction and particle activation, whereby the structure of the sample is broken. Coal breaks into separate organic components but with an active particle surface and many free organic radicals. A typical ball mill is similar to a cavitator in its principle of operation. It consists of a hollow ceramic cylinder, grinding balls of the same material, and specialized rolls. Before grinding, the initial coal (or coal processing waste), oils, distilled water and grinding balls are weighed. Coal and balls are poured into the cylinder (the coal to balls ratio for homogenization with extra mechanical action is usually 1:1). Water and different combustible liquids are added after that. The cylinder is placed on the rolls and the grinding begins (and lasts for one to two hours). The main difference of CWSP preparation using a ball mill from that using a homogenizer is the mechanical action of the grinding balls on the samples. This changes their rheological properties and ignition characteristics (due to a decrease in particle size).

Vershinina *et al.* [23] show that the most preferable method of CWS and CWSP production on the industrial scale is the use of cavitators or ball mills. For laboratory experiments, however, a homogenizer is enough. Similarly to [23], an MPW-324 homogenizer was used in this work.

The CWSP properties

Based on the research findings analyzed in overview [19], a reasonable conclusion could be made that the main properties of CWS and CWSP fuels can be defined by the properties of their components according to the additive rule (*i. e.*, proportional to the concentration of components with known properties). This principle was used to define the properties of CWSP fuels, tab. 6.

Component concentrations were varied for each CWSP composition for determination influence of this factor on the rheological properties of fuel. A wetting agent (plasticizer) was added to fuel compositions to provide a longer stability of CWSP properties. Table 7 presents the main characteristics of plasticizer.

Experimental set-up and procedures

The rheological properties of CWSP were measured by a rotary viscometer MLW Rheotest. The temperature of the sample was maintained by a water circulation thermostat MLW U15. A Searle-type coaxial cylinder system was used to determine the shear stress *vs.* shear rate. Shear viscosity for CWSP (filter cake of non-baking coal 49.5%, water 40.5%, fuel oil 10%) was calculated as a function of shear rate, fig. 1: $\eta = \tau/\gamma \cdot 100$. The relative error did not exceed 4%.

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Table 6. Properties of CWSP under study

Relative mass concentrations of CWSP components	Moisture, [%]	Ash, [%]	Viscosity, [mPa·s]	Stability, [day]	$Q^a_{s,V}$, [MJkg^{-1}]
Filter cake of flame coal 55%, water 45%	45	20.34	595	3	10.59
Filter cake of gas coal 55%, water 45%	45	18.60	365	3	12.20
Filter cake of coking coal 55%, water 45%	45	14.55	230	4	13.66
Filter cake of low-caing coal 55%, water 45%	45	27.99	403	3	8.38
Filter cake of non-baking coal 55%, water 45%	45	11.66	192	3	14.81
Filter cake of flame coal 49.5%, water 40.5%, oil 10%	40.5	18.31	480	7	14.04
Filter cake of gas coal 49.5%, water 40.5%, oil 10%	40.5	16.74	250	8	15.49
Filter cake of coking coal 49.5%, water 40.5%, oil 10%	40.5	13.09	269	7	16.80
Filter cake of low-caking coal 49.5%, water 40.5%, oil 10%	40.5	25.19	519	8	12.05
Filter cake of non-baking coal 49.5%, water 40.5%, oil 10%	40.5	10.49	365	8	17.84
Filter cake of non-baking coal 49.5%, water 40.5%, fuel oil 10%	40.5	11.11	269	9	17.27

Table 7. Main characteristics of plasticizer

Parameter	Value
Appearance	Transparent liquid
Surfactant content, [%wt.]	25
pH of the solution	6.5
Density at 293 K, [kgm^{-3}]	954
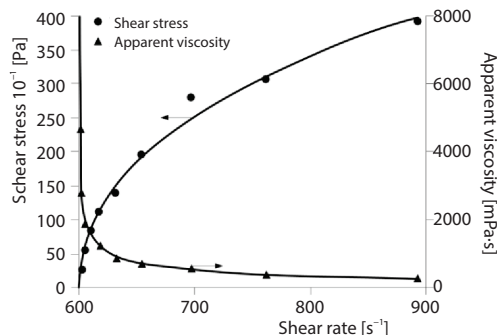
Figure 1. Shear stress and viscosity *vs.* shear rate for CWSP; dots are experimental data and curves are calculations by Herschel-Bulkley model

The determined shear stress and rate for all the compositions under study are adequately described, fig. 1, by a Herschel-Bulkley model. It is widely used to describe the rheological properties of compositions containing coal and liquid fuel components, *e. g.*, in [24]:

$$\tau = \tau_0 + k\gamma^n.$$

Results and discussion

Effect of coal processing waste properties

Figure 2 presents the viscosity of CWS based on processing wastes of coals of different ranks (shear rate 145.3 s^{-1}) *vs.* the H/C ratio in the cake. The H/C ratio characterizes the rank of the source coals. The analysis of this curve shows that if the component composition is the

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same, the viscosity of CWS based on cakes is primarily defined by the degree of coalification of the coal being processed. This conclusion correlates with the research findings in [25, 26]. The experiments in [25, 26] established that the surface of low-rank coal particles with a wide range of oxygen-containing groups is hydrophilic. With the increasing rank, coal gradually loses functional groups containing oxygen and becomes hydrophobic. The viscosity of CWS based on low-rank coals with $H/C{\sim}0.8$ is increased because coal particles are highly hydrated due to the hydrophilic carboxyl and hydroxyl groups, which form hydrogen bonds with polar water groups.

A reduction in the viscosity of CWS based on filter cakes made of coals with H/C < 0.7 stems from the conversion of coal structure in the course of metamorphism. As a result of this conversion, polar oxygen-containing groups dissolve with an increase in the share of aromatic and hydtroaromatic fragments. These structural changes in coals make them more hydrophobic (less wettable).

Adding a liquid fuel component to a CWS (and thus turning it into a CWSP) drastically changes its viscosity. At first glance, the viscosity vs. coal rank curve does not keep its shape, fig. 2. However, the analysis of viscosity variation from CWS to CWSP allows us to make a conclusion about a clear trend of viscosity change in the metamorphism series, fig. 3. Adding a petrochemical to CWS leads to increasing in the viscosity of filter cakes based on higher rank coals. Presumably, this results from intermolecular interactions between the hydrocarbons of oil and the hydrophobic surface of coal particles (the van der Waals forces). The particles become oily and agglomerate. Adding a petrochemical to the cakes based on lower rank coals, on the contrary, reduces the CWS viscosity. This may be conditioned by the disruption of molecular interactions between water molecules and the hydrophilic surface of coals, because the hydration shell of coal particles is partially destroyed, making them more hydrophobic.
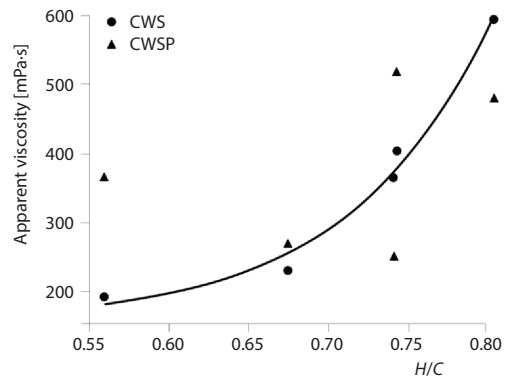

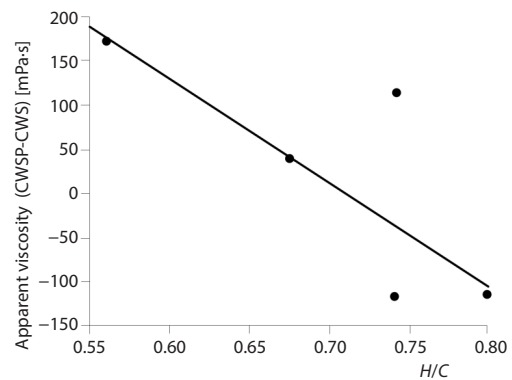**Figure 2. Calculated shear viscosity of CWS and CWSP *vs. H/C* ratio**

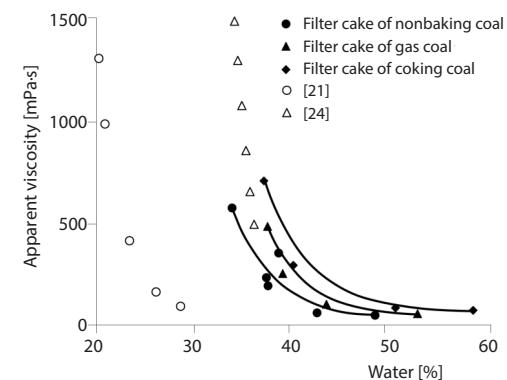**Figure 3. Viscosity variation *vs. H/C* ratio curve when switching from CWS to CWSP**

Figure 4. Shear viscosity of CWSP *vs.* water to coal component ratio (the proportion of turbine oil remained the same 10%)

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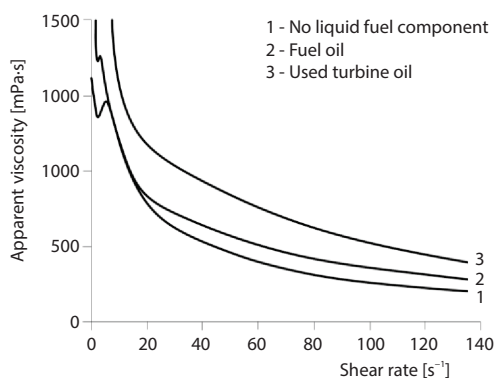
*Effect of water/solid coal
component concentration*

Figure 4 shows viscosity of CWSP based on the filter cake of non-baking coal (shear rate 145.3 s⁻¹) vs. water content. As you can see from fig. 4, the viscosity of CWSP declines exponentially with the increasing water content. The same shape of the curves is presented in [24, 27]. The water content in CWSP has a decisive effect on the rheological properties. This parameter makes it possible to regulate the viscosity of compositions in a wide range. At the same time, the main parameter characterizing all the fuels is the heat of combustion, which in our case will decline proportionally with the increasing water content in CWSP [13]. Therefore, the optimum water concentration in fuel slurry provides the adequate heat of combustion and acceptable viscosity for the practical use of CWSP on the actual coal-handling equipment.

In the case of comparable component compositions (in terms of moisture content and coal/liquid fuel component content), the viscosity of the fuels under study is comparable to the data presented in [27]. For example, for the fuel based on the filter cake of non-baking coal (composition: 36% of water, 54% of cake, 10% of oil), the viscosity equals to 576 mPa·s, and for the fuel of comparable composition described in [27], the viscosity is approx. 620 mPa·s. However, since various authors use components with a wide range of properties and quite an extensive variety of component compositions, quantitative characteristics may differ significantly as well, fig. 4.

Munoz *et al.* [28] give the maximum viscosities of 250-400 °C at 37.8 °C for pipelined heavy and extra-heavy oil. These values may serve as a reference for the viscosity of usable CWSP compositions.

*Effect of type and concentration
of combustible liquid*

In our experiments, fuel oil and used turbine oil served as liquid fuel components. Figure 5 shows the calculated viscosities of CWS and CWSP compositions based on filter cakes of non-baking coal with turbine oil and fuel oil. The introduction of a liquid fuel component generally makes a slurry more viscous. The greatest increase in viscosity comes with the use of turbine oil. The introduction of fuel oil leads to the medium values of viscosity. This behavior of the compositions stems from the individual characteristics of the liquid fuel component (*e. g.*, presence of certain functional groups) affecting the interaction of the liquid fuel component with the surface functional groups of solid components.

An increase in the concentration of the liquid fuel component (with the water to solid proportion remaining stable) leads to a practically linear increase in the viscosity of the composition, fig. 6. A similar rise in the viscosity induced by the introduction of liquid fuel components is described in [27]. With an increase in the share of the liquid fuel component, the combustion heat of the composite fuel will grow as well, whereas the ignition delay time and complete combustion time will short-

Figure 5. Calculated shear viscosities of CWSP with the introduction of liquid fuel components; the water to coal component ratio is constant; the content of liquid fuel component is 10%
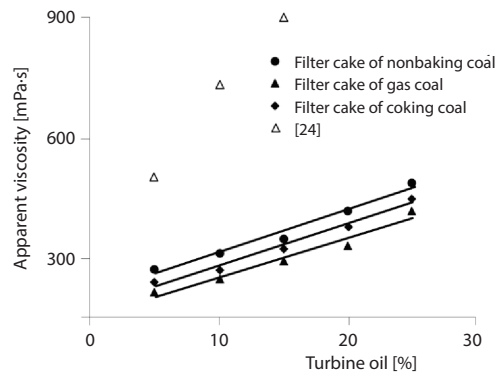
en [13]. As reflected in tab. 6, 10% of turbine oil added to the composition enables a 20-40% increase in the heat of combustion. This makes it possible to obtain the optimum CWSP parameters with due consideration of viscosities for various power plants.
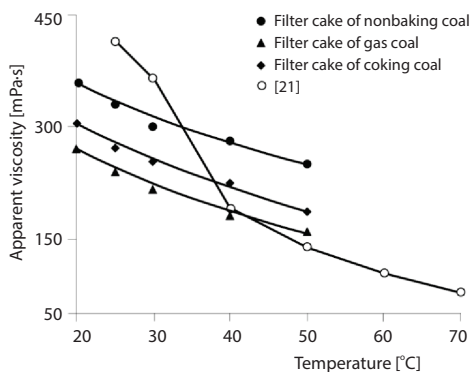
Effect of storage time and conditions

The dependences of the CWSP viscosity on the storage temperature were determined. Figure 7 shows the curve of the viscosity of CWSP based on the filter cake of non-baking coal (shear rate 145.3 s^{-1}) *vs.* the temperature of the composition. The viscosity of the composition goes down with increasing temperature;

Figure 6. Shear viscosity of CWSP *vs.* concentration of additional liquid fuel component

and if at 18 °C the viscosity is 370 mPa·s, by 50 °C it reaches 250 mPa·s. This shape of the viscosity *vs.* temperature curve is typical of such fuels and is described in many studies, *e. g.,* [24]. The temperature dependence of viscosity should be taken into account when planning the application of these fuels at actual power-generating facilities.

Furthermore, changes in the CWSP viscosity were studied in conditions of a long-term storage. For that purpose, fuel compositions were placed into a sealed container and kept it still in a laboratory at room temperature for as long as it was necessary. To determine the rheological properties, a sample from the lower part of the container was taken. While the compositions are stored, their viscosity increases significantly. Figure 8 presents the curve of viscosity *vs.* storage time of CWSP compositions. At the initial stage (~24 hours), the viscosity of the compositions grows rapidly. Later on, the growth of viscosity slows down and approaches the steady-state value. The rheological properties were investigated during the storage times of about a week. According to the forecast, the viscosity will continue to grow under longer storage conditions. The CWSP lamination has also been observed at the early stage with a small amount of water appearing on the surface. It is advisable to consider this behavior of viscosity if ready-made compositions are going to be stored before combustion in boilers. Otherwise, it is necessary to provide constant mixing of fuel in the containers or make the fuel less viscous in the first place.

Figure 7. Shear viscosity of CWSP *vs.* temperature (proportion of turbine oil 10%)
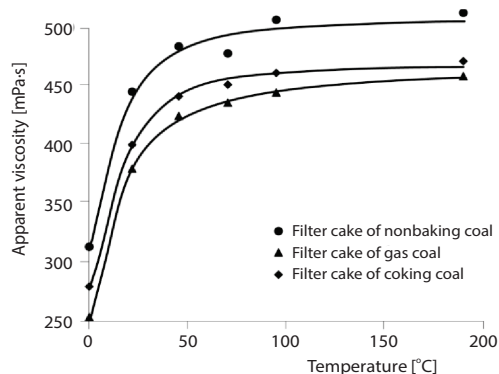
Figure 8. Shear viscosity of CWSP *vs.* storage time (proportion of turbine oil 10%)

Overall, the variation patterns of the rheological properties of fuels are in qualitative agreement with the properties described in literature for similar fuels. However, it is difficult to perform a quantitative comparison of fuel characteristics, since the number of possible components used by different authors is very large and the proportions of components in the compositions differ greatly as well. Therefore, this work is the first one to provide the database for CWSP preparation from coal and oil processing wastes.

For the first time, the rheological properties of fuel slurries based on coal and oil processing wastes were studied. The data obtained enable us to assert that such fuel compositions are fit for practical application in heat and power industry. Today, the world is accumulating vast and growing amounts of coal and oil processing wastes. For some regions, this is becoming a pressing issue, resulting, for example, in anthropogenic landslides on spoil banks of Kemerovo Region coal strip mines and the deterioration of the overall environmental situation. Therefore, waste recovery is a major and critical challenge.

Conclusions

- In this research, the rheological properties of promising CWSP have been determined. Such fuels based on coal processing wastes and used petroleum products, such as filter cakes of coal preparation and used industrial oils. The viscosity of the compositions under study ranges from 200-600 mPa·s.
- The rheological properties of waste-based CWSP compositions are as good as (or even better than) those of CWS or CWSP fuels based on high-rank coals and eligible for pipe-line shipment.
- The established effects of the decisive factors (properties and types of components, storage time and conditions) on the rheological characteristics of CWSP fuels are the main database for the predictive modelling of the processes involved in the preparation, transportation and usage of CWSP in heat and power industry.

Acknowledgment

Nomenclature

A^d – ash level of dry sample, [%]

C^{daf} – fraction of carbon in the sample converted to a dry ash-free state, [%]

H^{daf} – fraction of hydrogen in the sample converted to a dry ash-free state, [%]

k – consistency parameters, [Pa·s^n]

N^{daf} – fraction of nitrogen in the sample converted to a dry ash-free state, [%]

n – index

O^{daf} – fraction of oxygen in the sample converted to a dry ash-free state, [%]

$Q_{s,V}^a$ – enthalpy of combustion of wet sample of coal, [Jkg^{-1}]

$Q_{s,V}^d$ – enthalpy of combustion of dry sample of coal, [Jkg^{-1}]

S_t^{daf} – fraction of sulfur in the sample converted to a dry ash-free state, [%]

T_f – flash temperature, [K]

T_{ign} – ignition temperature, [K]

V^{daf} – yield of volatiles of coal to a dry ash-free state, [%]

W^a – humidity of analytical sample of coal in an air-dry state, [%]

Greek symbols

γ – shear rate, [s^{-1}]

η – shear viscosity, [Pa·s]

ρ – density, [kgm^{-3}]

τ – shear stress, [Pa]

τ_0 – yield stress, [Pa]

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