About determining the wettability of coal surface

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Abstract. A new method of measuring the wettability of coal powders based on determining the mass fraction of wetted powder particles at their interaction with water droplets during the process of gravitational sedimentation in a transparent cell has been proposed. Wettability parameter is calculated from the results of measuring the dependence of spectral transmittance of laser emission in the cell on time of powder sedimentation.

1. Introduction.

The effectiveness of modern methods of coal preparation and processing is achieved by using new energy and environmentally beneficial technical and technological solutions based on background knowledge about the phenomena of chemical and physical and chemical processes taking place on the interfaces (liquid - solid - gas). In particular, in many processes of coal-mining industry the data on the wettability process and contact angle value of coal surface are required. As an example, the process of separating the minerals from draw rock during the coal flotation process at coal benefication [1]. Coal wettability (hydrophobicity or hydrophility) is one of the main parameters that determines the process quality. In the technology of wet dust extraction in coal mines including the water pulverization in the cloud of suspended coal particles the data on powder particles wettability is required for effective particles «enveloping» by liquid and removing them from the air flow.

Since coal is an organic sedimentary rock with a complex structure the question of determining its wettability is complex and controversy. The basic method of determining the wettability of the coal surface is the method of "sitting" drop [2] that consists in determining the contact angle of a drop placed on the surface of the sample. In literature there are three basic ways to prepare the working surface of the coal sample. The first method is based on the briquetting of coal powder particles in tablets [3], the second one is to use a monolithic coal sample [4], and a third method consists in grinding the coal monolith [5].

This paper describes a method for determining the wettability of the fine coal powder without any mechanical action on the surface of coal particles that allows to perform the measurement in a dusty cloud directly [6].

2. Method of measurement.
The proposed method for determining the wettability of the fine powders is based on calculating the fraction of wetted powder particles. A uniform air-born dust is created in a cell with transparent plate walls. The maximum diameter of particles is no more than 5 μm. Using a laser and radiation detector the spectral transmittance of dust suspension is measured (fig. 1). Then, a system of monodispersed droplets with diameter 0.8÷2.5 mm from droppers that is placed uniformly on cross-section of the cell is given in the cell. After that, the spectral transmittance is measured again. The initial concentration of powder particles is taken from the condition $T_0 \leq 0.2$ (where $T_0$ is the spectral transmittance before supplying the droplets) and time interval of droplets supplying is determined from the condition $T_k > 2T_0$ (where $T_k$ is the spectral transmittance after supplying the droplets).

![Figure 1. Scheme of experimental setup for determining the wettability of fine powder: 1 – cell; 2 – air-born dust; 3 – laser; 4 – radiation detector; 5 – system of droplets supplying; 6 – liquid](image)

The wettability characteristic is assumed the wettability parameter [2] – mass fraction of dust particles settled on a droplet (wetted particles)

$$\beta = \frac{m_c}{m},$$

where $m_c$ is the mass of wetted dust particles (settled on a droplet); $m$ is the mass of powder particles that run against the droplet on its free settling in the cell.

In the cell with a height $h$, with a droplet of a diameter $D$, $m$ is calculated

$$m = \frac{\pi D^2}{4} h c \eta,$$

where $c$ is the mass concentration of powder particles in the cell; $\eta \leq 1$ is the capture coefficient.

Mass of wetted powder particles taking into account (1) is equal to

$$m_c = \beta m = \beta \frac{\pi D^2}{4} h c \eta.$$  

The analysis of powder particle settling on a moving droplet is to take into account curving of dust flow lines. The capture coefficient is a ratio of particles that run against the droplet to particles
that could run against the droplet if their flow lines never skirted the droplet. As a consequence, not all particles placed in cross-section\( S = \pi D^2 / 4 \) (midlength section of the droplet) run against the droplet. The share of collided particles is determined (for potential flow-around) from the Langmuir-Blodgett formula\[7\]

\[
\eta = \left( \frac{\text{Stk}}{\text{Stk} + 0.125} \right)^2 ,
\]

where \( \text{Stk} \) is Stokes number.

For monodispersed powder particles of a diameter \( D_p \), Stokes number is determined using the equation

\[
\text{Stk} = \frac{\rho_p D_p u}{18 \mu D} ,
\]

where \( \rho_p \) is the density of particle; \( u \) is the droplet velocity; \( \mu \) is the coefficient of dynamic viscosity of liquid.

Since a air-born dust is a system of polydispersed particles it is necessary to use average Stokes number\[7\]:

\[
\text{Stk} = \int_0^\infty \rho_p D_p^2 \varphi(D_p) u \frac{dD_p}{18 \mu D} = \frac{\rho_p u}{18 \mu D} \int_0^\infty D_p^2 \varphi(D_p) dD_p = \frac{\rho_p u}{18 \mu D} D_{20} ,
\]

where \( \varphi(D_p) \) is the differential function of counting size distribution of powder particles; \( D_{20} = \int_0^\infty D_p^2 \varphi(D_p) dD_p \) is the mean-square diameter of powder particles.

\( n \) drop bottles are arranged in upper part of the cell to give the flow of monodispersed droplets (fig. 1). The drop bottles are placed in cross-section. All drop bottles form a droplet of equal diameter \( D \) at the frequency \( f \) (number of generated droplets per second). Therefore, on a time interval \( t \), \( N \) droplets pass the cell:

\[
N = n ft .
\]

Since the mass of powder particles settled on a drop is determined using the formula (2), for \( N \) droplets the sum mass of wetted particles is equal to

\[
M_c = \beta \frac{\pi D^2}{4} hcnf \eta = \beta \frac{\pi D^2}{4} hcnf \eta .
\]

It is necessary to take into account the time change of mass concentration of powder particles in the cell to determine the wettability parameter \( \beta \) since some share of particles is settled on droplets. For this reason the equation (4) is written as

\[
dM_c(t) = \beta \frac{\pi D^2}{4} hcn(t) nft \eta dt ,
\]

where \( dM_c(t) \) is the mass of wetted particles in the time \( dt \).

The particles wetted and droplets are settled on the bottom of the cell so the reduction in sum mass of suspended particles \( M(t) \) is:

\[
dM(t) = -dM_c(t) .
\]

Taking into account (6) the equation (5) has the form

\[
dM(t) = -\beta \frac{\pi D^2}{4} hcn(t) nft \eta dt .
\]

Dividing each member of the above equation by the cell volume \( V \) yields:
Equation (7) can be written in the form
\[ \frac{dc(t)}{c(t)} = -\beta B dt. \]  
(9)

Integrating (9) in the range of \( t = 0 \) to \( t \) allows:
\[ \ln \left\{ \frac{c(t)}{c_0} \right\} = -\beta B t. \]

It follows that
\[ c(t) = c_0 \exp(-\beta B t), \]
(10)

where \( c(t) \) is the mass concentration of powder particles at a time \( t > 0 \); \( c_0 \) is the initial mass concentration of powder particles.

From the equation (10) the wettability parameter \( \beta \) can be defined as:
\[ \beta = \frac{\ln[c_0/c(t)]}{B t}. \]
(11)

Placing (11) in the equation for \( \beta \) from equation (8), we obtain:
\[ \beta = \frac{4V \ln[c_0/c_k]}{\pi D^2 \eta c k}, \]
(12)

where \( c_k \) is the concentration at time \( t_k \) corresponding to water droplets outage.

It is necessary to determine the mass concentration of powder particles in the cell in initial time \( c_0 \) (beginning of the droplets supplying) and after settling of the droplets \( c_k \) to determine the wettability parameter \( \beta \) using the formula (12).

For this reason a uniform air-born dust is created in the cell with plate parallel transparent walls (for example, glass). Using the source of probe radiation (laser) and radiation detector the spectral transmittance in the cell is measured
\[ T = \frac{I}{I_0}, \]

where \( I \) is the intensity of radiation passed through suspended particles; \( I_0 \) is the intensity of radiation of incoming beam.

In accordance with Bouguer’s law [8]
\[ T = \exp(-\tau), \]
where \( \tau = Kc l \) is the spectral optical density of powder particles layer; \( K \) is the spectral index of depletion that characterizes the reduction of light intensity by unit volume of medium including independently scattering particles; \( l \) is the cell width (the thickness of the powder layer).

For polydisperse particles with distribution function \( \varphi(D_p) \) the index of depletion is [8]
\[ K = \frac{3c}{2 \rho_p} \left\{ \int_0^{\infty} Q(\alpha, \bar{m}) D_p^2 \varphi(D_p) dD_p \right\}, \]
where \( Q(\alpha, \bar{m}) \) is the efficiency factor of depletion; \( \alpha = \pi D_p / \lambda \) is the dimensionless diffraction parameter (Mi parameter); \( \lambda \) is the wave-length of probing radiation; \( \bar{m} \) is the complex refractive index of the material of the particles.

Assuming that the distribution function \( \varphi(D_p) \) does not change during the settling of the powder particles on droplet, we can write

\[
c_0 / c_k = \tau_0 / \tau_k = \ln \left( \frac{T_0}{T_k} \right).
\]

where \( \tau_0, \tau_k \) is the spectral optical density of powder layer before and after droplets settling, correspondingly.

Placing (13) in (12) we obtain the working formula for calculating \( \beta \)

\[
\beta = 4V \ln \left[ \ln \left( \frac{T_0}{T_k} \right)^{-1} \right] / \pi D^2 h n f \eta.
\]

3. Example of realization of the method.

The possibility of realization of the method has been determined by means of performing direct calculations of change of concentration and optical density of medium for coal dust at their settling in an air at giving the water droplets for parameters presented in Table 1. The distribution function of the coal particles was obtained using the setup Mastersizer 2000 (MALVERN, UK). Rms particle diameter is \( (D_p)_{20} = 1.9 \mu m \). The obtained distribution function was approximated by a gamma distribution

\[
\varphi(D_p) = 15.9 \ D_p^{5.5} \ exp(-3.6D_p),
\]

where \([\varphi(D_p)] = \mu m^{-1} ; \ [D_p] = \mu m \).

Table 1. Values of parameters for calculation of \( \beta \)

| Parameter | Value |
|-----------|-------|
| \( \rho_p \) | 1200 kg/m³ |
| \( \mu \) | 1.808·10⁻⁵ kg/m·s |
| \( n \) | 50 |
| \( f \) | 2 s⁻¹ |
| \( \rho \) | 1.205 kg/m³ |
| \( h \) | 0.2 m |
| \( V \) | 0.002 m³ |
| \( \eta \) | 0.53 |
| \( \rho_l \) | 1000 kg/m³ |
| \( D \) | 1.5 mm |
| \( (D_p)_{20} \) | 1.9 \( \mu \) m |
| \( t_0 \) | 100 s |

The results of calculation are presented in fig. 2, 3. The dependence of dimensionless concentration of suspended coal particles \( \overline{c}(t) = c(t)/c_0 \) (where \( c \) is the current value of the concentration of particles in the cloud) on time of droplets supplying for the wettability parameter value \( \beta = 0.8 \) is given in fig. 2. Dependence of optical density on time of droplets supplying in the cell at value \( \beta = 0.8 \) is presented in fig. 3. Check the adequacy of the method can be carried out using fig. 2 and fig. 3.

Select the time interval of droplet feed \( t_0 = 100 s \) at which the change of the initial concentration of suspended powder is significant and the condition \( T_i > 2T_0 \) for spectral transmittance occurs (fig. 2, 3). At time values \( t_0 = 100 s \) the concentration of suspended particles and ratio of optical densities are equal to \( \overline{c}_k = 0.47, \ \tau_0 / \tau_k = 2.1 \), correspondingly.

Placing found values in the formula (14) for calculating the parameter \( \beta \), we obtain \( \beta = 0.8 \).
As we can see from the above example, specified and calculated values of wettability parameter coincide with each other (β = 0.8). Similar results are obtained for any value of the wettability parameter in the range β = 0÷1.0.
4. Experimental working out the method

Working out the method for measuring the wettability of coal powder is carried out using the experimental setup (fig. 4) comprising of the cell, system for creating of suspended powder, system for droplets supplying in the cell, and system for measuring the spectral transmittance.

System for monodispersed droplets supplying – nine of uniformly placed drop bottles – is arranged in upper part of the cell by size of 10×10×20 sm and with walls from optical glass. The variation of the diameter of the capillary and the height of the liquid column in the drop bottle provides to obtain a stream of droplets of distilled water of the spherical shape and predetermined size \( D = 1 \div 2.5 \text{ mm} \) with a predetermined frequency of separation \( f = 0.5 \div 5 \text{ Hz} \).

System for creating the uniform suspended coal particles includes the source of compressed air and tube placed along lateral and bottom side of the cell. The specified mass of coal powder is placed in the ell to obtain the cloud of suspended particles. Air is fed through the holes in the tube, located at the bottom of the cell. During the air supplying, the cell is closed with lid and suspended coal particles cloud is created.

System for measuring the spectral transmittance includes the source of probing radiation - helium-neon laser LG-78 with power of 1 mW and a wavelength of \( \lambda = 0.6328 \mu \text{m} \) and radiation detector - germanium transistor FTT-4 with an area of the sensor element of 3 mm\(^2\). The radiation detector is located in light-tight housing with pinhole aperture on its butt end to exclude the effect of background illumination. Measurement of spectral transmittance is performed at condition of passing the probing radiation on the distance 12 sm from upper end of the cell. Register signal from a radiation detector is carried by a digital oscilloscope GDS-2064 agreed upon with personal computer.
The visualization of the particle cloud is conducted using the video camera located vertical to probing beam of the laser to control of uniformity of distribution of coal particles. Working out the method is conducted for coal samples. The characteristics of the samples (maximum particle diameter $D_{\text{max}}$, moisture content $W$, ash content $Z$, volatile content $V^{\text{daf}}$) are given in Table 2.

| Sample | $D_{\text{max}}$, mm | $W$, % wt | $Z$, % wt | $V^{\text{daf}}$, % wt |
|--------|----------------------|-----------|-----------|----------------------|
| 1      | 0.05                 | 0.4       | 21.86     | 23.72                |
| 2      | 0.05                 | 0.4       | 7.0       | 24.13                |
| 3      | 0.05                 | 0.5       | 17.05     | 33.63                |

The results of working out the method allowed finding the procedural error of experimental setup for realization of the method for determining the powder wettability. In particular, during the atomization of coal powder by compressed air some of the particles located on the cell walls. It results in the error in measuring the spectral transmittance. Relevant modification of experimental setup is necessary to create a uniformly distributed particle cloud isolated from the inner cell walls.

5. Conclusion
A method for determining the wettability of the fine coal powder has been proposed. The proposed method allows improving the accuracy of determining the characteristics of the wettability of the fine powder and carry out the measurement directly in a dusty cloud. This method can be used to study a broad class of organic and inorganic powder materials. As a result of working out the experimental setup, the difficulties associated with the creation of a uniform cloud of suspended particles that is isolated from the inner cell walls have been found.

6. References
[1] Abramov A A 2008 *Moscow State university*. Flotation methods of beneficiaction.
[2] Zimon A D 1974 *Moscow. Chemistry*. Liquid adhesion and wettability.
[3] Li Man Xu Hai-Hong Shu Xin-qian 2008 *Journal of coal science and engineering*. Study on coal dust wettability measurement using cold briquetting technique. Vol. 14 № 4 pp. 571-574
[4] Arkhipov V A, Korotkikh A G, Patrakov Yu F, Usmaina A S 2015 *Journal Tuva Staty university. Tekhnicheskie and fiziko-matematicheskie nauki*. Experimental study of the effect of method of sample preparation on the coal surface wettability. Vol 3. pp. 95-104
[5] Drelich J, Laskowski J S, Pawlik M 2000 *Coal Preparation*. Improved sample preparation and surface analysis methodology for contact angle measurements on coal (heterogeneous) surfaces. Vol. 21 № 3 pp. 246-275
[6] Arkhipov V A, Zmanovskiy S V, Paleev D Yu, Patrakov Yu F, Usanina A S 2014 *The patent of the RF* 2522805. Method for determining the fine powder wettability / Publ. 20.07.2014, № 20
[7] Shilyaev M I, Shilyaev A M 2003 *Publishing house Tomsk State University of Control Systems and Radioelectronics*. Air dynamics and heat-and-mass transfer of gas dispersed flows.
[8] Arkhipov V A, Bondarchuk S S 2010 *Tomsk State University*. Optical methods of diagnostics of heterogeneous plasma of combustion products.