

## Studying the Physical Properties of Coal-Oil Concentrate

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### **Abstract**

The article includes comprehensive studies of the physical properties of certain structural types of coal-oil concentrate. We conducted a review and analysis of established and innovative methods for studying the strength, stickiness and hydrophobic properties of the surface of coal-oil aggregates. We reviewed and analysed the methods for assessing the mechanical and penetration strength of individual coal-oil aggregates, the integral mechanical strength of coal granulate using the "cylinder-punch" method, as well as substance plastic strength of coal-oil aggregates. We obtained a quantitative assessment of the following physical properties of certain types of coal-oil concentrate structures: strength using the penetration method; plastic strength of the aggregate substance; stickiness to fluoropolymer; the fraction of the aggregate surface covered with a hydrophobic binder. It has been found that the studied types I, II, III and IV of coal-oil aggregate structures exhibit significant differences in physical properties, which evidently defines their essential technological properties.

A promising way for further research involves exploring how the structural characteristics of coal-oil agglomerates affect their technological properties, particularly as objects of dehydration, hydraulic transportation, combustion in furnaces, coking, pyrolysis, and as carriers in processes for adhesive enrichment of hydrophobic minerals. This approach will allow identifying the most promising application areas for coal aggregates of various structural types, formulating the primary consumer requirements for their properties, establishing an a posteriori basis for finding optimal technological aggregation modes in each specific process application, and delineating the main directions for necessary research in each field.

**Keywords:** *Selective oil agglomeration of coal; Coal-oil concentrate; Physical properties; Mechanical; Penetration and plastic strength; Stickiness; Hydrophobic properties; Structural types of coal-oil aggregates.*

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## **1. Introduction**

Selective oil agglomeration of coal is a specialized process aimed to enrich and dewater fine coal in water-hydrocarbon-coal mixtures, which includes selective wetting of coal particles with hydrocarbon liquid, intensive (turbulent) mixing of the water-coal-oil mixture facilitating active adhesive interaction between hydrocarbon components, resulting in the formation of coal-oil structures. During the turbulization process of suspension, primary coal-oil aggregates undergo structural transformation into agglomerates and dense spherical granules. Hydrophilic mineral particles remain in the aqueous phase of the suspension and are separated along with it from the granular concentrate in the dewatering units.

The final product of oil agglomeration of coal is the oil-coal concentrate, either agglomerate or granulate.

The most well-known technologies for selective oil agglomeration of fine coal, developed since the early 20th century, include: the Trent Process [1]; the "Convertol" processes [1-3]; CFRI (Central Fuel Research Institute, India) [1, 4-6]; NRCC National Research Council Canada [7-8]; Shell method (Shell Oil SPS technology Shell Pelletizing Separator) [1, 4]; the BHP method (Broken Hill Proprietary, Australia); the "Olifloc" process (Germany) [1, 9]. Several variations of oil agglomeration of coal have been developed in Ukraine for hydraulically transported coal enrichment residues, coking and steam coal [10-11]. Despite the long-established history of oil agglomeration of coal, both scientific and industrial interest in it remains strong [12-15]. Whereas one of the least studied issues is the physical, chemical and technological properties of coal-oil concentrate. Studying them and comparing the results is complicated by the diversity of the raw material base, which includes coal at various metamorphism stages, and basin-specific coal properties influenced by coal formation conditions, coal petrography, geochemical composition of mine waters. All these characteristics affect the aggregation ability of the coal substance and, ultimately, the quality of the coal-oil concentrate. In addition, both the process of coal oil aggregation and the physical, chemical and technological properties of the coal-oil concentrate are significantly influenced by another component – the binding reagent, which can be a wide range of substances such as petroleum products, liquid coke products, secondary oils.

The analysis of the existing array of experimental data and theoretical works on selective oil aggregation of coal showed that without a clear classification of coal aggregates by key features, theoretical statements are prone to ambiguity to say the least, while technological recommendations in various scenarios may prove to be erroneous. Pursuant to the reasoning presented in several studies [1, 20-21], the classification of coal-oil concentrate has been proposed to be based on the structural characteristics of coal-oil aggregates. The justification for it arises, firstly, from the fact that the structure of the aggregates is influenced by all input and operational parameters of the coal-oil aggregation process. Secondly, it is the structure of the units that determines their technological properties. Thirdly, the structure of aggregates is determined by the type and correlation of aggregate-forming forces [20-22]. Therefore, it serves as a comprehensive integrating feature that reflects both the combined impact of various factors on the technological process and the outcomes of the process, enabling conclusions to be drawn about the mechanism of aggregate formation.

In the study [1], based on the synthesis of research results and approximately 200 polished sections of coal-oil agglomerates and granules the following main types of structures were identified (Figure 1): I – condensed formations characterized by the presence of thin boundary films of binder between individual grains; II – structures with concave menisci of binding material between coal grains on the aggregate surface; III – droplets of binding material filled with coal grains; IV – loose formations (clusters) of coal grains bound by bridges of binding substance.



Figure 1. Main types of structures of coal-oil aggregates. The illustrations were prepared from observations through the eyepiece of MBS-9, NEOPHOT-21 microscopes.

Therefore, it is relevant to investigate the physical properties of the highlighted main types of structures of coal-oil aggregates. The goal of this study is to investigate the physical properties of certain types of structures of coal-oil concentrate. The objectives of the study are to review and analyse the methods of investigating the strength, stickiness and lyophilic (hydro-

phobic) properties of the surface of coal-oil aggregates and to conduct a quantitative assessment of the following physical properties of certain types of structures of coal-oil concentrate: strength using the penetration method; plastic strength of the aggregate substance; stickiness to fluoropolymer; the fraction of the aggregate surface covered with a hydrophobic binder.

## 2. Experimental

### 2.1. Methods and materials

The coal from the Donetsk coal basin of grade G (gas) with a grain size of 1-0 and 0.1-0 mm was used to produce coal-oil aggregates of several structural types, along with mazut M100 as the primary binding agent. The pelletization process coal-oil aggregates was carried out at a laboratory installation with turbulent mixing of water-coal-oil mixture. The process operating parameters were selected according to the recommendations [1], individually for each specific structural type of coal-oil concentrate.

### 2.2. Research methods

#### 2.2.1. The assessment of mechanical strength of coal-oil aggregates

The mechanical strength of an individual aggregate granule or a sample of up to 24 pieces (biologically strong aggregates obtained, for example, using a binder like polymer from benzene production in coke-chemical or oil refining plants) can be assessed by subjecting it to static linear loading and recording the force at the point of failure. For this purpose, it is appropriate to use a type strength tester such as the PK-21-0.15, designed to test catalysts, sorbents and other granular materials for mechanical strength under static conditions by compression. The measuring range is from 1.5 to 150 N, with an average error of 1%. If necessary, the PK-21-3.0 tester can be chosen for the same purpose, which possesses the strength measurement range from 30 to 3000 N, with failure force determination error of 1%.

#### 2.2.2. The assessment of strength of coal-oil aggregates using the penetration method of Donetsk National Technical University (DonNTU)

DonNTU has developed an original device, designed by Yu. L. Papushyn, specifically for assessing the strength of individual aggregates (granules), based on the penetration method (Figure 2).

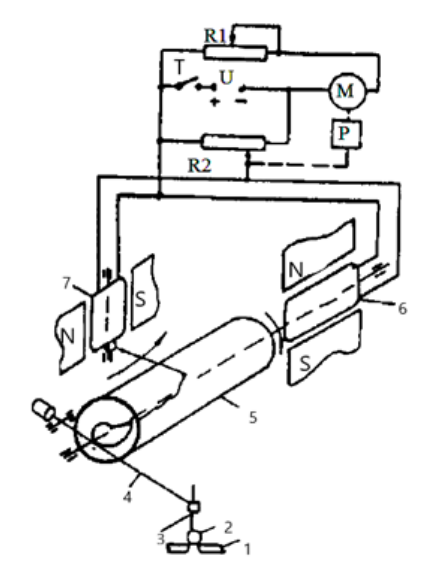


Figure 2. The principal diagram of DonNTU penetration device for determining the strength of granules: 1 – object table, 2 – test granule, 3 – indenter, 4 – lever, 5 – drum, 6, 7 – drum and recording mechanism frames.

The functions of the indenter are performed by a metal needle with a diameter of 0.3 mm with a flat end. The device includes a mechanical stress generating unit (micro-electric motor M, gearbox P, resistors R<sub>1</sub> and R<sub>2</sub>, power supply U), an object table and a mechanical loading system. The aggregate loading process is recorded automatically on the diagram with "force *P* – displacement *l*" coordinates. The numerical value of the destructive force (tensile strength) is determined by the point where the tangent line intersects the *P(l)* curve in the region of maximum indenter displacement speed. The device allows for determining the tensile strength of the aggregates during their brittle and plastic fracture in the range of 0 to 8 N, for aggregates with diameter up to 20 mm, adjustable within 1-10 mm/min loading speed, with an error of ±0.1 N. In single measurements, the indenter needle can be replaced with a flat loading surface, thereby expanding the capabilities of the device.

### 2.2.3. The assessment of strength of coal-oil aggregates using the "cylinder-punch" method

The "cylinder-punch" method can be utilized to assess the integral mechanical strength of coal granules. A calibration device is coaxially mounted on the working cylinder (Figure 3). A granulate sample is poured into the cylinder cavity without compaction and shaking.

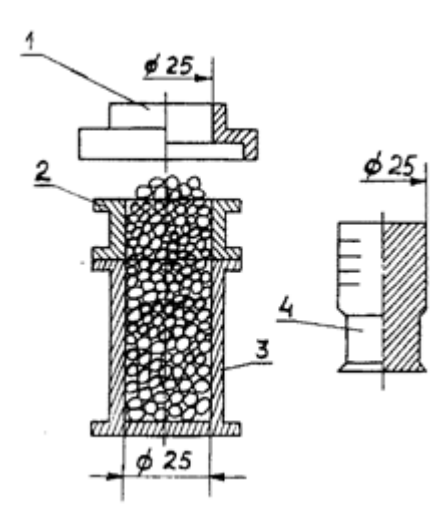


Figure 3. Granulate strength determining device: 1 – guide sleeve, 2 – calibration device, 3 – cylinder, 4 – punch.

The calibration device is then removed by sliding it off ("cutting" along the flange). A guide sleeve is installed on the cylinder into which the punch is lowered until it touches the material. Assembled in this manner, the device is installed between the piston and the press cushion. After this, the punch is gradually pressed to a fixed depth (10 mm) at a speed of 1 mm/s. The manometer gauge reading of the press is recorded. With the dimensions of the device as shown in Figure 3, pressing the punch to a depth of 10 mm corresponds to compressing the granule sample by 20% of its volume. The results of determining the mechanical strength of the granules are expressed as the compressive strength limit of the granule layer:

$$\sigma_{ct} = \left( \frac{D_n}{d_n} \right)^2 P_c \quad (1)$$

where  $P_c$  is the average manometer gauge reading in kg/cm<sup>2</sup>;  $D_n$  is the piston diameter;  $d_n$  is the punch diameter.

We have proposed a modified method for determining the mechanical strength of granulate, which consists in loading a portion of granules filled into the cylinder cavity with a constant weight while using a piston. In this case, the strength is assessed based on the change in volume  $\Delta V_r$  of the granulate expressed in % (or based on the contraction  $\Delta l_r$  in %).

#### 2.2.4. The assessment of plastic strength coal-oil aggregate substances

The plastic strength of a granulation (agglomeration) substance is measured by a plastometer, for example, type K - 01 (Figure 4) designed to determine the strength of coagulation structures of paste-like materials.

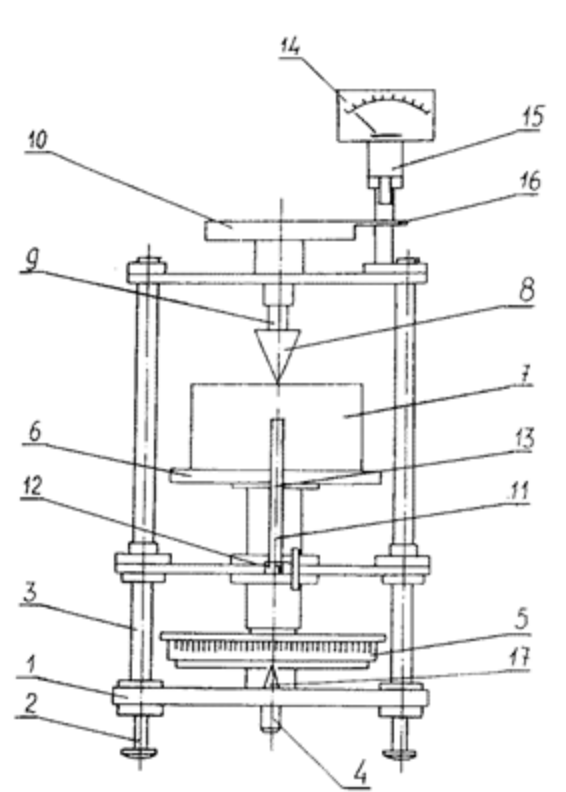


Figure 4. Type K-01 plastometer, general layout: 1 – base, 2 – leg, 3 – stand, 4 – shaft, 5 – nut-disc with limb, 6 – table, 7 – basin, 8 – cone-indenter, 9 – rod, 10 – platform, 11 – ruler, 12 – clamp, 13 – arrow, 14 – clock-type indicator, 15 – holder, 16 – beam, 17 – arrow.

The device is based on the principle of penetration of the loaded cone into the test mass. As the area is penetrated, the shear stress decreases until it balances with the plastic strength of the mass structure [16]. The value of the plastic strength of a pre-homogenized and compacted granulation substance before the elastic effect appears is determined by the following formula:

$$P_m = K_\alpha \frac{F_k}{h_n^2} \quad (2)$$

where  $K_\alpha$  as a constant dependent on the cone angle  $\alpha$ ; when  $\alpha = 45^\circ$   $K_\alpha = 0.41$  [17].  $F_k$  is the load on the cone determined by the weight of the load;  $h_n$  is the depth of cone penetration.

The key characteristics of the K-01 plastometer are: the measurement range of plastic strength of  $P_m = (2.6 \cdot 10^3 - 8.5 \cdot 10^5) \text{ N/m}^3$ ; the error  $\sigma_{pm}$  not exceeding 10%; range of load changes on the cone of  $F = (1.4 - 101.4) \text{ N}$ ; total penetration depth of the cone of  $h_{ok} = 0 - 15 \text{ mm}$ ; working depth of  $h_{pk} = 7 - 15 \text{ mm}$ .

#### 2.2.5. The assessment of coal-oil aggregates stickiness

The stickiness of coal-oil structures can be determined by the empirical formula of Yelishchevych [18]:

$$S_f = \frac{P_s \times \tau_{fs}}{S_{pl} \times \tau_{ds}} \quad (3)$$

where  $P_s$  as the force causing delamination;  $S_{pl}$  as the area of the plate;  $\tau_{fs}$  as the delamination duration;  $\tau_{ds}$  as the delamination duration for ultimately deteriorated structure of oil binding agents.

Given the low spinability of oil agents (where spinability refers to the ability to produce long thin threads under certain conditions) used in the processes of oil aggregation of coal, the formula (3) can be simplified by assuming  $\tau_{fs} \cong \tau_{ds}$ . However, there is still ambiguity in the definition of stickiness, due to the dependency of measurement outcomes on the speed of force application (plat separation). This is highlighted, in particular, by N. Debroyn and R. Guvink [19]. Therefore, V. Biletskyi proposed to determine the stickiness by the following formulas [11]:

$$S_d = \int_{\tau_{fs}}^{\tau_{max}} \frac{P_s}{S_{pl}} \quad (4)$$

where  $\tau_{max}$  as the value of  $\tau_{fs}$  at which  $P_s \rightarrow 0$ .

At this approach, the parameter  $S_f$  corresponds physically to the area under the curve  $P_s / S_{pl} (\tau_{fs})$ . By analogy with viscosity and in order to distinguish it from the parameter  $S_f$ , it is convenient to call the parameter  $S_d$  *dynamic stickiness*, which fully corresponds to its physical meaning.

To measure  $S_f$  and  $S_d$ , we constructed a special device similar to the one described in [18]. The basis for it was technical (laboratory) scales. A measuring vessel with 1 mL divisions was installed on one scale, positioned opposite and lower to a water container and a control valve connected to a drain tube. Instead of the second scale, a counterweight and a plate made of the material under study were suspended. Prior to measuring the stickiness, the plate was carefully bonded to the coal-oil substance of the agglomerate or granulate.

The fraction of aggregate surface covered with a hydrophobic binding reagent was determined using the selective dye sorption method (method of Kolbanovska [23]).

### 3. Results and discussion

The results of experimental determination of coal-oil aggregate strength using the penetration method; the plastic strength of the aggregate substance; its stickiness to fluoropolymer; the fraction of aggregate surfaces covered with a hydrophobic binding reagent are presented in Tables 1 and 2.

In practice, coal-oil concentrates of structural types I, II and IV are most frequently encountered. Consequently, we conducted an additional assessment of the fraction of the coal-oil aggregate surface covered with a binder, following the method of A. S. Kolbanovska (Table 3).

Table 1. The results of experimental studies of coal-oil aggregates of various structural types for strength using the penetration method.

| Coal-oil concentrate structure type | Initial coal size <sup>1</sup> | Mass fraction of mazut in aggregates <sup>2</sup> , % | Aggregates diameter <sup>3</sup> $d_a$ , mm | Strength of aggregates by the penetration method $P_p$ , $10^{-3}$ N |
|-------------------------------------|--------------------------------|---|---|--|
| I                                   | 0 - 1.0                        | 1 - 3   | 0.3 - 1.2                                   | 2.5 - 3.3  |
|                                     | 0 - 0.1                        | 5 - 7   | 0.2 - 0.4                                   | 2.5 - 3.0  |
| II                                  | 0 - 1.0                        | 8 - 15  | 0.5 - 3.0                                   | 1.6 - 1.9  |
|                                     | 0 - 0.1                        | 20 - 25   | 0.5 - 3.0                                   | 1.4 - 1.7  |
| III                                 | 0 - 1.0                        | over 20   | 1 - 5                                       | 1.2 - 1.4  |
|                                     | 0 - 0.1                        | over 30   | 1 - 5                                       | 1.1 - 1.3  |
| IV                                  | 0.3 - 1.0                      | 4 - 5   | 0.4 - 2.0                                   | -  |

<sup>1</sup>According to the screen analysis; <sup>2</sup>According to reagent dosage during the preparation of the initial water-oil-coal mixture; <sup>3</sup>According to microscopic evidence (MBS-9 microscope)



Table 2. The results of experimental studies of coal-oil aggregates of various structural types for plastic strength, stickiness and the fraction of aggregate surface covered with binder.

| Coal-oil concentrate structure type | Initial coal size <sup>1</sup> | Plastic strength of the aggregate substance <sup>1</sup><br>$P_m$ , kg/cm <sup>2</sup> | Stickiness to fluoropolymer <sup>2</sup><br>$S_f$ , g/cm <sup>2</sup> | Fraction of the granule surface covered with mazut <sup>3</sup><br>% |
|-------------------------------------|--------------------------------|--|---|--|
| I                                   | 0 - 1.0                        | 1 - 3  | 0.3 - 1.2   | 2.5 - 3.3  |
|                                     | 0 - 0.1                        | 5 - 7  | 0.2 - 0.4   | 2.5 - 3.0  |
| II                                  | 0 - 1.0                        | 8 - 15   | 0.5 - 3.0   | 1.6 - 1.9  |
|                                     | 0 - 0.1                        | 20 - 25  | 0.5 - 3.0   | 1.4 - 1.7  |
| III                                 | 0 - 1.0                        | over 20  | 1 - 5   | 1.2 - 1.4  |
|                                     | 0 - 0.1                        | over 30  | 1 - 5   | 1.1 - 1.3  |
| IV                                  | 0.3 - 1.0                      | 4 - 5  | 0.4 - 2.0   | -  |

<sup>1</sup> According to the results of studies using the K-01 plastometer; <sup>2</sup> According to the method of Yelishevych [18]; <sup>3</sup> According to the method of Kolbanovska [23]

Table 3. Fraction of the surface area of the aggregate coal grains with an oil reagent (% of the area of direct "coal-oil" contact: % of the area of contact through the aqueous film).

| Coal-oil aggregate type                               | M100 mazut | Kerosene | Anthracene oil | AAP-2 floatation reagent |
|---|------------|----------|----------------|--------------------------|
| Granules, $Q_M=20$ wt%<br>structure type II           | 81:14.7    | 78:19.8  | 87:10          | 86:5.12                  |
| Agglomerates, $Q_M=5$ wt%<br>structure types I and IV | 79:0       | -        | 83:2           | 85:0                     |

Thus, the studies indicate that coal-oil aggregates of structural type I are characterized by a minimum total binder content (1-3 wt% for coal with a size of 0 - 1.0 mm and 5-7 wt% for coal with a size of 0 - 0.1 mm), almost complete absence of binder in the bulk state, and binding of coal grains by oil films with a thickness of 1-3  $\mu$ m (polished sections study observed on NEOPHOT-21 microscope), maximum plastic strength of the aggregate substance, low stickiness and incomplete coverage of the aggregate surface with the oil reagent and almost complete absence of coal grain coating by the binding reagent on the aqueous film. A distinctive feature of most type I granules and agglomerates is a clearly defined "core-shell" structure, which helps to preserve the coking properties of coal in hydraulic transport conditions [24, 25].

Type II coal-oil aggregates occupy an intermediate position between types I and III aggregates. These aggregates exhibit an average plastic and penetration strength, maximum but unstable stickiness dependent on the fraction of binder in the aggregate, and are almost completely covered with an oil binder (for mazut, it ranges from 86 to 95% of the surface). At the same time, 5-20% of the coal surface is covered with a aqueous film binder reagent (Table 3).

Type III aggregates contain the binder mainly in its bulk state. The plastic and penetration strength of these aggregates is minimal, the stickiness is average, but stable, and the entire surface is covered with an oil phase.

A characteristic feature of type IV structures is a bridging connection between coal grains. These may include liquid bridges, and bridges made of hardening or paste-like binders. These structures are characterized by low plastic strength, low stickiness, and a low degree of reagent coverage of the aggregates. Additionally, there is almost no coal surface covered with the binding reagent on the aqueous film (Table 3).

Based on the conducted research and the data from [1, 20-21], we can conclude that the aggregate-forming forces in type II structures are primarily capillary forces on the aggregate surface, in type III structures, they are represented by the surface tension force of binder droplets, while in types I and IV these are the adhesion forces through oil films and binder bridges, respectively.

#### 4. Conclusions

For the first time in the practice of selective oil aggregation of coal, this study has reviewed and analysed both well-known and original methods for investigating the strength, stickiness and lyophilic (hydrophobic) properties of the coal-oil aggregate surfaces. In particular, the methods for assessing mechanical and penetration strength of individual coal-oil aggregates, the integral mechanical strength of coal granulate using the "cylinder-punch" method, and the plastic strength of coal-oil aggregate substances have been reviewed and analysed.

Comprehensive investigations have been conducted and a quantitative assessment has been obtained for various physical properties of individual types of coal-oil concentrate structures, such as: strength using the penetration method; plastic strength of the aggregate substance; stickiness to fluoropolymer; the fraction of the aggregate surface covered with a hydrophobic binder. The results reveal that the investigated coal-oil aggregate structures of types I, II, III and IV have significant differences in physical properties, which evidently defines their essential technological properties.

A promising area for future research involves examining how the structural characteristics of coal-oil agglomerates influence their technological properties. This includes their roles as objects of dewatering, hydraulic transportation, combustion in furnaces, coking, pyrolysis, and as carriers in processes for adhesive enrichment of hydrophobic minerals. This approach will allow identifying the most promising application areas for coal aggregates of various structural types, formulating the primary consumer requirements for their properties, establishing an a posteriori basis for finding optimal technological aggregation modes in each specific process application, and delineating the main directions for necessary research in each field.

#### Symbols

|        |  |
|--------|--|
| CFRI   | <i>Central Fuel Research Institute;</i>      |
| NRCC   | <i>National Research Council Canada;</i>     |
| BHP    | <i>Broken Hill Proprietary;</i>              |
| DonNTU | <i>Donetsk National Technical University</i> |

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