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IDENTIFICATION OF THE MECHANISM OF POWDERY MATERIALS GRANULES FORMATION DURING WET GRANULATION IN A HIGH-SPEED ROTARY GRANULATOR

A. E. Lebedev[1], A. A. Murashov[2], I. S. Gudanov[1], A. A. Vatagin[1], D. S. Dolgin[1], F. S. Orlov[1]

Anton Evgenievich Lebedev, Doctor of Technical Sciences, Professor of the Department of Technical Machines and Equipment, [2]**Anatoly Alexandrovich Murashov,** Doctor of Technical Sciences, Professor of the Department of Mathematics; **Ilya Sergeevich Gudanov**, Candidate of Technical Sciences, Head of the Department of Technical Machines and Equipment; **Alexander Alexandrovich Vatagin,** Candidate of Technical Sciences, Associate Professor of the Department of Technical Machines and Equipment; **Dmitry Sergeevich Dolgin**, Senior lecturer; [1]**Fedor Sergeyevich Orlov**, Postgraduate student.

[1]Yaroslavl State Technical University, 88 Moskovsky prospekt, Yaroslavl, 150023, Russia, *lae4444@mail.ru, goudanov@yandex.ru, vatagerr@bk.ru, dim_dol.94@mail.ru, orlov.frgu@yandex.ru*

[2]Yaroslavl Higher Military School of Air Defence named after Marshal of the Soviet Union L. A. Govorov, 150001, Russia, Yaroslavl, Moskovsky prospekt, 28, *alena.severyanka@mail.ru*

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Abstract. *The article concerns with the mechanism of carbon black granules formation during wet granulation by adding a special liquid binder additive to the dusting carbon black. The research describes a design of a vertical type laboratory granulation unit consisting of a drive, tripod, and frame. The main element of the frame is a shaft with pins mounted on it. As a result, we developed a methodology for conducting the experiment. The article provides a graph of the dependence of bulk density and power on the time of the process. It determines the following stages of the granulation process: wetting of dusting carbon black, granulation transition, stage of intensive granulation changes, stabilisation of granulation changes. In industrial granulators every stage has a distribution in accordance to the apparatus length. All these stages have different power characteristics.*

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Introduction

Granulation is one of the most effective ways of compacting powdered materials. It is widely used in transport, mining, and construction engineering [1, 2]. This process is one of the most important stages in the production of carbon black (CB). Due to it CB commercial form is obtained – spherical granules with a diameter of 0.5-2.0 mm [3].

Carbon black is a dark-colored powder formed in the gas phase during thermooxidation or thermal pyrolysis of hydrocarbon raw materials. It accompanied by the decomposition of hydrocarbons into carbon and hydrogen under the high temperature. The main consumer of CB is the tire and rubber industries; CB is used as an active filler, especially in synthetic rubbers [4-9].

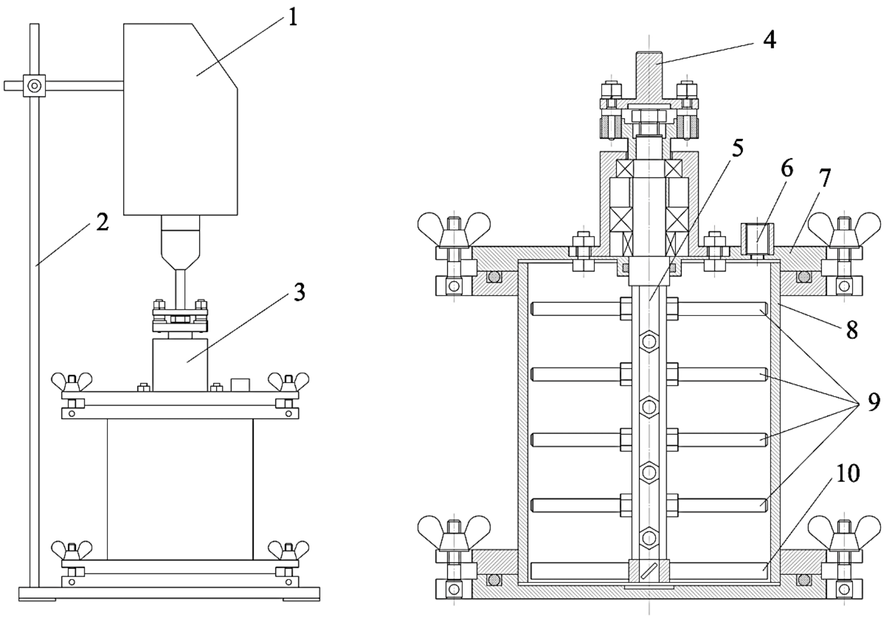
Currently, high-speed granulators with a rotating finger shaft are used in industry to produce granules by wet granulation method [4, 10-15]. It concerns with the introduction of binder component aqueous solution into the dusting CB.

Research problem statement

However, the widespread use of the granulation method does not provide improving the quality of commercial technical specifications. Moreover, there is no appropriate theory of wet granulation, and the mechanism of granule formation. Therefore, it is almost impossible to improve the efficiency of granulators, reduce the consumption of water and the binder component, increase the uniformity and quality stability of the resulting granular product in terms of bulk density, strength characteristics of granules, and granulometric composition. Identifying the mechanism of granule formation will address the problems mentioned above and improve the quality of finished products.

Materials and methods

To identify the mechanism of CB wet granulation and provide the formation of CB wet granulation theory, a pilot unit was developed. The scheme is shown in Fig. 1.



1 -drive; 2 -tripod; 3 -laboratory granulator; 4 -pin flexible coupling; 5 -shaft; 6 -pipe for feeding the binder solution; 7 -top cover; 8 -frame; 9 -fingers; 10 -paddle agitator

Fig. 1. Scheme of the laboratory unit

The following fixed parameters were adopted:

- CB – N660 in dusty form;
- the ratio of the binder additive solution to the dusting CB is 120 ml/100 g;

- the concentration of the aqueous solution of the binder additive is 0.2% by weight;
- the rotation speed of the mixer is 400 rpm;
- the gap between the frame and the rotor fingers is 2 mm.

For the initial dusting CB of the N660 grade, the bulk density was determined in accordance with the method [10] and was 146 kg/m^3.

Experimental Part

We loaded 100 g of dusting CB into the granulator; the granulator rotor is placed inside the frame and fixed with clips on the top lid; 120 ml of a binder additive solution of a given concentration is poured into the frame; the agitator is turned on and the time from the start of operation is set. After the set time the motor is switched off; the rotor is taken out; the obtained product is poured into a metal tray and sent for drying in a drying chamber at a temperature of 120 °C for 24 h. After drying for the obtained product, a bulk density in accordance with the methods is determined [4, 10-15].

CB samples were run at 5-second intervals for the duration of the granulation process up to the moment of stabilisation of the product bulk density – further at 10-second intervals. For the purpose of statistical data processing, 3 samples were prepared for each time interval. The arithmetic average of the results of determining product bulk density corresponding to a certain duration of the process was taken as the result of measuring the parameters for 3 samples.

The experiment was conducted in the vertical position of the granulator. This approach to the implementation of the experiment simulates the process in each individual zone of the horizontal apparatus. Indeed, the process time for the vertical position conditionally corresponds to the length of the horizontal granulator. Moreover, the implementation of the process in a vertical position avoids the formation of stagnant zones in the granulator.

Results and Discussion

The graph in Fig. 2 clearly demonstrates the stages of the granulation process. Each stage is characterised by individual dependences of bulk density on process duration. They can be described as close to linear ones.

At the first stage of the process (up to 35 s inclusive), there is a linear increase in the bulk density of the material. The dependence of the current load on the agitator at the beginning of this stage is also characterised by a linear increase with reaching a plateau at the end. After the rotor stops, a multiphase system is observed at each point of the first stage – dusty CB with the potential presence of small-sized "germ" granules, an interphase layer consisting of waterlogged CB, and an aqueous solution of a binder additive, indicating the further proceeding of the wetting process.

At the second stage (from 35 to 40 s) of the process, the effect of a "granulation transition" is observed – the system inside the granulator completely transits into a single-phase (granule-like) state with an increased number of large fragments (Fig. 3). This effect shows the end of the wetting process – the aqueous solution of the binder additive completely passes into the associated state.

The dependences of the current load on the agitator and the bulk density of the material in this area indicate the abrupt and short-term nature of this transition. One of the explanations for the "granulation transition" is the achievement of a critical content of small-sized "germ" granules in the mass of the material. The use of the existing method of analysing the granulation composition does not allow us to determine the content of such granules – sieves are clogged with a large amount of dusty CB. Therefore, it is necessary to develop a special method for analysing the granulation composition with an emphasis on determining the content of small granules with a high content of dusty CB.

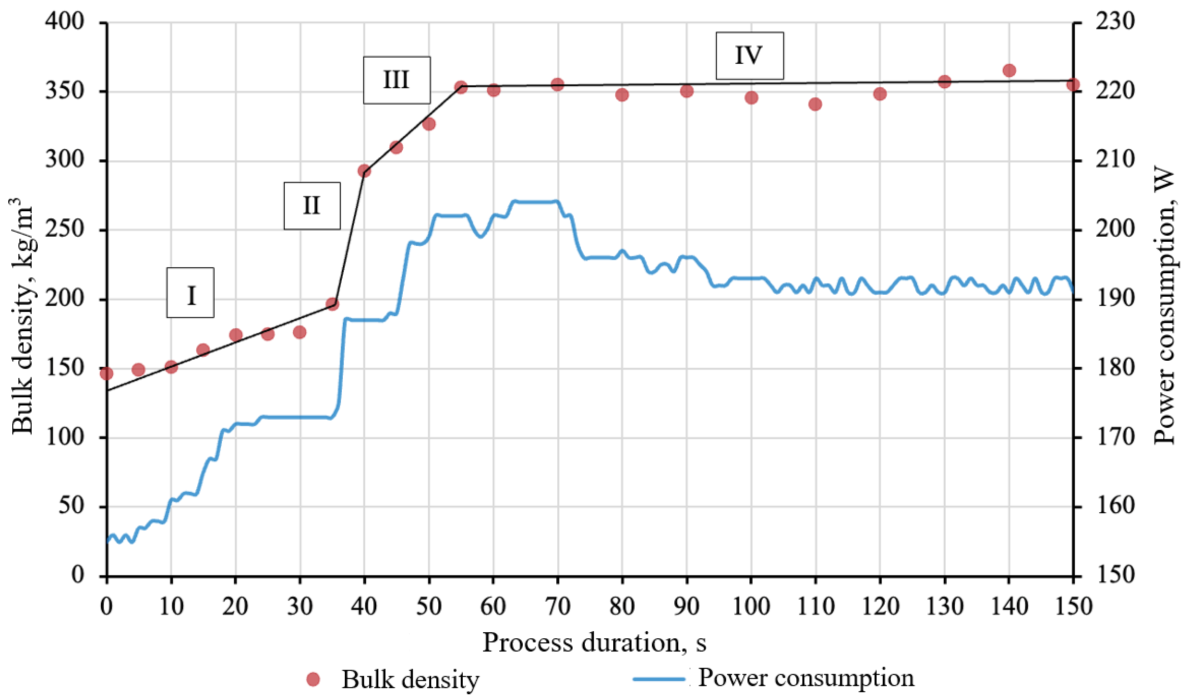


Fig. 2. The dependence of the bulk density of the samples and the current load on the agitator on process duration

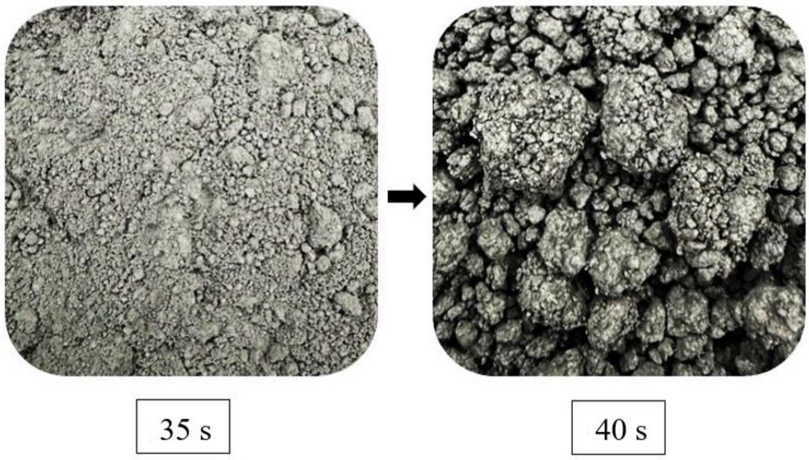


Fig. 3. Changing the structure and shape of the granular mass at the second stage of the process

The effect of the "granulation transition" is also confirmed by a sharp increase in the current load on the agitator, resulting from a sharp increase in the resistance of the material during the transition to a granular form.

However, detailed examination of the granulation mass obtained at 40 c shows the structure of large fragments as the clumped granules of smaller sizes.

At the third stage (from 40 to 55 s) of the process, there is a decrease in the intensity of bulk density increase. Provided the wetting process is completed in stage II, the change of this characteristic together with the increase of lustre on the granule surface indicate the processes of granule compaction and grinding, redistribution of granulometric composition through the destruction of larger fragments, and reduction of the average diameter (Fig. 4). The observed increase in the current load on the agitator in this time interval is due to a change in the characteristics of granules subjected to fracture, abrasion, and grinding. As the process proceeds, progressively less uncompacted brittle granules remain in the system. The sources for such changes are increasingly stronger granules, requiring more energy to deform if the intensity of granulation redistribution within the system is maintained.

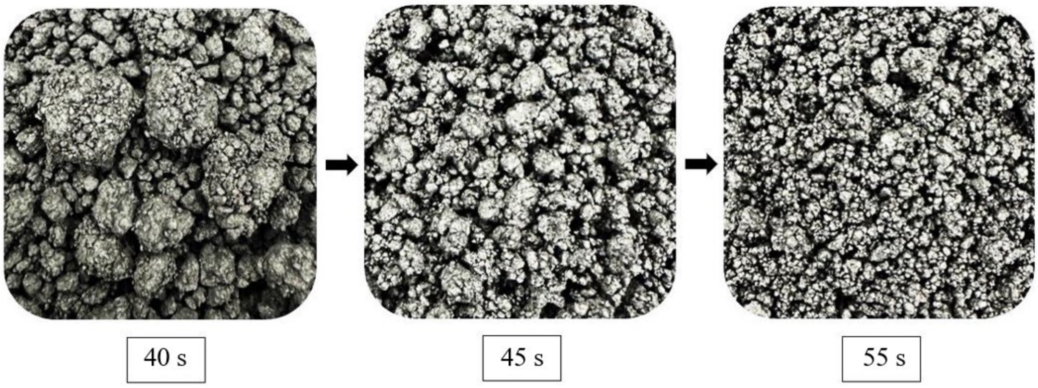


Fig. 4. Changing the structure and shape of the granular mass at the second stage of the process

The final stage IV is not definitively interpreted. On the one hand, the value of the bulk density, according to the approximating line, increases as the process proceeds. On the other hand, this increase does not exceed the convergence value for the standard method for determining this indicator. The current load on the agitator is reduced to the stabilisation of this indicator from 95 C. The absence of significant visual changes in the material (Fig. 5), changes in bulk density and current load on the agitator indicates stabilisation of the system inside the granulator. Therefore, the intensity of granulation changes is significantly reduced.

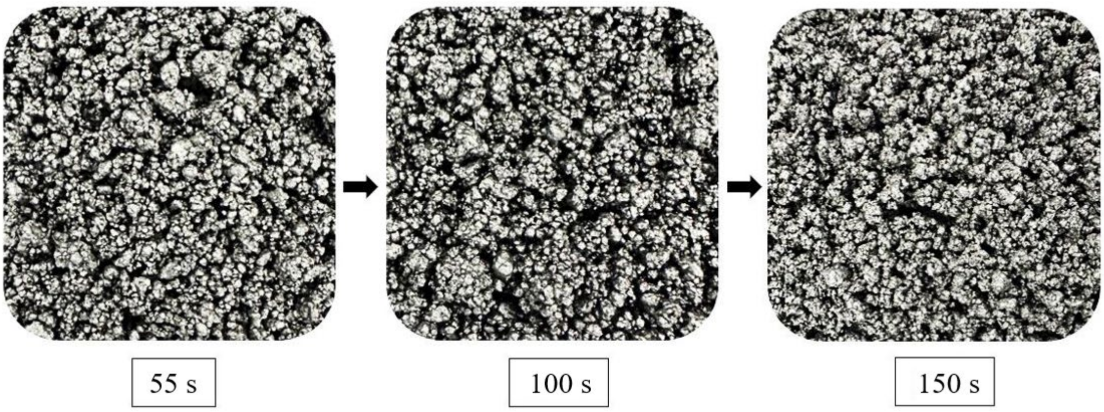


Fig. 5. Changing the structure and shape of the granular mass at the IV stage of the process

Conclusions and Recommendations

As a result of the conducted research, the process of granule formation occurs in four stages as follows:

1. Wetting of the dusty CB.
2. Granulation transition.
3. The stage of intense granulation changes.
4. Stabilisation of granulation changes.

In industrial granulators every stage has a distribution in accordance to the apparatus length. All these stages have different power characteristics.

References

1. **Orlov V.Yu., Komarov A.M., Lyapina L.A.** Production and use of technical carbon for rubber. Yaroslavl: Alexander Rutman Publishing House, 2002, 512 pp. (in Russian).
2. **Kugatov P.V., Ivashkina E.A., Zhirnov B.S.** Granular Carbon Adsorbent Based on Carbon Black and Synthetic Pitch. *Solid Fuel Chem.*, 2023, 57, 423-427. DOI: 10.3103/S0361521923060034.
3. **Makarenkov D.A., Nazarov V.I., Mavlyudova Ya.A.** Features of the granulation process of fuel compositions by rolling on a rotary granulator. *Vestnik MGOU. Seriya: Yestestvennyye nauki [Bulletin of Moscow State Open University. Series: Natural Sciences]*, 2015, 1, 42-49. (in Russian).
4. **Sevostyanov V.S., Apatenko A.S., Shamgulov R.Yu., Protsenko A.M.** Development of technical means for processing and granulating technical carbon using thermolysis technology. *STIN [Machines and Instrument]*, 2022, 42(**5**), 538-540. DOI: 10.3103/S1068798X22050240. (in Russian).
5. **Igumenova T.I., Shulga A.M.** Problems of using pyrolysis carbon black in tire production: materialy LVIII otchetnoy nauch. konf. prepodavateley i nauchnykh sotrudnikov Voronezh State University of Engineering Technologies. Ch. 1. Voronezh: Izd-vo VSUET, 2020, P. 104. (in Russian)
6. **Zhuravskii G.I.** Technical carbon from the products of thermolysis of discarded tires. *IFZH [Journal of Engineering Physics and Thermophysics]*, 2020, 93(**6**), 1470-1475. DOI 10.1007/s10891-020-02252-y. (in Russian).
7. **Ivanovsky V.I.** Carbon black. Processes and apparatuses. Omsk: OAO Tekhuglerod, 2004, 228 pp. (in Russian)
8. **Vasiliev P.S., Pavlova A.E.** Improvement of the equipment design of the stage of granulation of technical carbon by the wet method. *Energo- i resursosberezhenie: promyshlennost' i transport [Energy and resource saving: industry and transport]*, 2023, 1(**42**), 11-19. (in Russian).
9. **Rodionov D.A., Suvorina I.V., Knyazev Yu.V.** Recycling of car tires. *Molodoy ucheniy [Young scientist]*, 2016, 4(**108**), 936-937. Available at: https://moluch.ru/archive/108/25962 (accessed: 09.03.2025).
10. **Shamgulov R.Yu., Goncharov A.N.** Development of a drum-screw unit for granulating carbon black. Materialy mezhdunar. nauch.-tekhn. konf. molodykh uchenykh Belorusskogo gosudarstvennogo technicheskogo universiteta im. V.G. Shukhova. Belgorod: Izd-vo BGTU im. V.G. Shukhova, 2021, P. 1844-1847. (in Russian).
11. **ASTM D1513-05(2023)**. Standard Test Method for Carbon Black, Pelleted – Pour Density. Annual Book of ASTM Standards, 2023, 09.01, 2 p.
12. **Bortnikov V.G.** Theoretical foundations and technology of processing plastics: textbook. Moscow: INFRA-M, 2015, 480 pp. (in Russian)
13. **Koltsova T.B., Tsobkallo E.S., Meshcheryakova G.P.** Modeling of Deformation Properties of Composite Material Filled with Technical Carbon. *FibreChem*, 2024, 56, 226-229. DOI: 10.1007/s10692-024-10559-2.
14. **Kolipaka S.S., Junqueira L.A., Ross S., Garg V., Hossein Mithu MdS.H, Bhatt S., Douroumis D.** An Advanced Twin-Screw Granulation Technology: The use of Non-Volatile Solvents with High Solubilizing Capacity. *AAPS PharmSciTech*, 2024, 25, 174. DOI: 10.1208/s12249-024-02890-y.
15. **Kugatov P.V., Ivashkina E.A., Zhirnov B.S.** Granular Carbon Adsorbent Based on Carbon Black and Synthetic Pitch. *SolidFuelChem.*, 2023, 57, 423-427. DOI: 10.3103/S0361521923060034.

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