ACCELERATION OF PRODUCTION PROCESS FOR PETROLEUM PITCH

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The production of anodes and electrodes from petroleum coke and petroleum-derived binders is one of the promising directions in improving the quality of graphitic carbon materials [1, 2].

Methods have been proposed for producing petroleum pitch by low-temperature thermocondensation of cracked tars; a rather long period, more than 3 h, is required to bring the quality indices of the residue up to the standards for electrode pitch (GOST 10200-62). Research showed that the process of pitch production could be accelerated by increasing the temperature and pressure in the reaction zone with subsequent removal of the light products at low pressures; i.e., the single-stage process for producing pitch by thermocondensation of the cracked tar is effected at a temperature of 460-500°C and a gage pressure of 15-35 kgf/cm²; in the second stage, the light fractions are driven off at 380-420°C and atmospheric pressure. This separation of stages makes it possible to select optimum conditions for each stage of the process in accordance with the type of raw material and the required pitch quality, and thereby reduce the time required to produce the pitch without reducing the yield.

The primary quality indices for electrode pitch are the softening point, content of volatile matter, and content of toluene-insoluble compounds. It is considered that the most desirable properties for electrode pitch are a softening point of 85-90°C, volatiles content of 55%, and toluene insolubles of 30-35% [3].

As a raw material for pitch production, we used a cracked tar from the Omsk Petroleum Refining Combine, of distillate origin, with a density of 1.1048 g/cm³ and a carbon residue of 23.2%.

The results from thermocondensation of the cracked tar by the accelerated method and by the previously proposed method are shown in Figs. 1-4. It will be seen from Figs. 2 and 3 that, as the polycondensation of the cracked tar is continued, the volatiles content decreases and the softening point increases. The changes in yield and properties of the polymerizing residue are caused by the accumulation of the asphaltic-resinous substances formed through chemical reactions of decomposition and polycondensation, as well as physical process of vaporization. By regulating the degree of conversion of the tar, its properties can be brought into line with the standards imposed on electrode pitch. The curves for the volatiles content tend to flatten out toward the end of the time intervals that were investigated, even though the curves for the yield of polymerized residue become steeper, thus indicating a retardation of the polycondensation process (see Fig. 1).

These same trends are observed in low-temperature thermocondensation (see Fig. 4). The relative locations of the curves in Figs. 1-4 depend on the temperature and pressure of the process. The yield of polymerized residue at temperatures of 460 and 480°C becomes greater when the gage pressure is increased from 15 to 35 kgf/cm², as this increase in pressure brings the intermediate decomposition products into the pitch formation process; naturally, this causes an increase in the time required for the polycondensation reactions. At 500°C, however, this relationship is no longer observed; the yield curve for the pitch residue at a gage pressure of 15 kgf/cm² is higher than that at 35 kgf/cm². This is explained on the basis that during the polycondensation, the heavy intermediate products that pass into the liquid phase are accompanied by low-boiling and less aromatic fractions that lower the solvency of the medium with respect to asphaltenes and thus promote aggregation of the asphaltenes. The aggregates subsequently undergo autonomous thermocondensation without any significant involvement of the intermediate decomposition products dissolved in the dispersion medium. Under these conditions, it should be noted, the intermediate products undergo primarily decomposition reactions. The light hydrocarbons that are formed are removed in the
Fig. 1. Influence of thermocondensation conditions on pitch yield: 1) 500°C, 15 kgf/cm² gage; 2) 480°C, 15 kgf/cm² gage; 3) 460°C, 15 kgf/cm² gage; 1') 500°C, 35 kgf/cm² gage; 2') 480°C, 35 kgf/cm² gage; 3') 460°C, 35 kgf/cm² gage.

Fig. 2. Influence of thermocondensation conditions on volatiles content of pitch. Curve Nos. same as in Fig. 1.

Fig. 3. Influence of thermocondensation conditions on softening point of pitch. Curve Nos. same as in Fig. 1.

Fig. 4. Yield and quality of pitch as functions of thermocondensation time at 420°C with gage pressure of 4 kgf/cm²: 1) pitch yield; 2) softening point; 3) volatiles content.