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PROPERTIES OF SEMI-COKING TAR PITCHES

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Abstract. *This paper compares previously produced pitches derived from medium-rank coal semi-coking tars. The study considers methods for obtaining semi-coking tars in different atmospheres. We measured pitch softening temperatures and compared how these values depend on both the original coal grade and tar extraction technology. We produced pitch by extracting tar from Zh-grade coal processed in an inert atmosphere. The carbonization experiments compared semi-coking tar pitch with grade B electrode pitch.*

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Introduction

Coal tar pitch (CP) is a residue of coal tar separation into fractions: light fraction (T<170°C); phenolic fraction (T = 170-210 °C); naphthalene fraction (T = 210-230 °C); absorption fraction (T = 230-270 °C); anthracene fraction (270-360 °C); coal tar pitch (T < 360 °C). The production of pitch from semi-coking tar (SCT) is of particular scientific interest. The following promising applications for SCT-derived pitches have been identified:

1. Materials for road construction [1].
2. Medium-temperature pitches as binders in electrode material production [2].
3. Binders for anode mass [3].
4. Binding fillers for coking charge blends [4].

The expanded potential and prospects for SCT pitch applications, the advancements in production technology ensure the relevance of this work. The experimental data obtained could significantly contribute to improving the processing of semi-coking byproducts. In this study, the melting point of previously obtained pitches will be measured and compared with their softening points. For the first time, pitch will be extracted from low-temperature pyrolysis tar of Zh-grade coal in an inert atmosphere, with its softening point, melting point, and technical analysis determination. A novel comparative analysis was conducted between pitches obtained via extraction (this work) and those previously isolated by distillation. Additionally, low-temperature carbonization (T = 550 °C) will be performed on both SCT-derived pitch and commercial electrode pitch. The uniqueness of these investigations defines the novelty of this work.

Experimental part

1. Review of methods for producing various pitches from SCT. This section presents the results of previous studies on the production of pitches from SCT. Moreover, the results of processing SCT from various raw materials (bituminous coals, lignites, oil shales, peat) are shown. Also, the paper provides the results of obtaining pitches from SCT using various extraction technologies.

The authors consider the technologies for producing pitches from bituminous coal SCT. In study [5], semi-cokes were obtained by semi-coking at T = 500–550 °C, and SCT was collected in this temperature range. The collected SCT was distilled up to T = 320 °C, resulting in pitches with T_s = 59 °C and 71 °C. The hydrogen content in the pitches was 7.55–7.44%; the carbon content increased from 79.8% to 82.8% with increasing Ts; the oxygen content was ~8% [5]. In study [6], bituminous coal SCT was distilled up to 360 °C; yielding 50% pitch; phenol content in the pitch of 11.3% (equivalent to 5.6% relative to the original SCT). In study [7], the distillation of bituminous coal SCT was obtained with the yield 78.6% pitch. Industrial SCT was subjected to batch distillation up to T = 390 °C, producing pitch with T_s = 56 °C and a 47% yield. Increasing the final temperature to T = 405 °C the authors obtained pitch with T_s = 65 °C [8]. In study [2], pitch with T_s = 75 °C (30% yield) was obtained from grade D coal SCT by thermal oxidation (TO) at T = 230–348 °C (duration 30 min, air flow rate 63 L/h). The fractional composition of the obtained pitch was determined as toluene-insoluble fraction (α) = 20%; quinoline-insoluble fraction (α_1) = 1.9%; coke residue = 30.2%; ash content = 0.04%. In study [9], pitch with T_s = 138 °C was obtained from industrial SCT by TO (T = 265–370 °C, 30 min, air flow 40 L/kg). In study [4], bituminous coal SCT was subjected to vacuum distillation up to T = 400–410 °C at a residual pressure of 2–3 kPa, yielding pitch with T_s = 159 °C and a 50% yield. According to [2, 4, 5, 8], soft pitches can be obtained by distilling bituminous coal SCT up to 360 °C. Electrode pitches, binder pitches, and high-temperature pitches are produced by TO or by increasing the distillation temperature of SCT above 400 °C. Analysis of studies [2, 4, 6–8] shows that the yield of pitch from bituminous coal SCT varied in the range of 30–78%.

Also, lignite SCT (coal grade B) is used as raw material for pitch production. In [10], semi-coking of lignite from the Lelchitsy deposit, the Gomel region, the Republic of Belarus was examined. The SCR yield during semi-coking (T = 550 °C) varied in the range of 3.4-23% [10]. During distillation of lignite SCT, pitch yields were 13.07%, 18.20%, and 37.59% [10]. In study [11], lignite SCT was distilled up to 350 °C, yielding 45.25% pitch. In study [12], fractions up to 250 °C were distilled from primary lignite tar, and the remaining portion (70% of total tar) underwent TO for 8 hours at T = 200 °C in the presence of a catalyst in the form of iron salts of organic acid (0.1% by weight of initial tar), ultimately producing high-temperature pitch. In [13], lignite SCT was distilled up to 350 °C, producing pitch with volatile matter content of 83.6%, ash content of 0.24%, and fractional composition: α = 12.6%, β = 71.3%, γ = 16.1%. On the experiments described above, distillation of lignite SCT at 360 °C mainly produces soft pitches, while the thermal oxidation method is used to obtain high-temperature pitch.

Shale SCR is of particular interest as a raw material for pitch production. In [3], binder pitches and electrode pitches were obtained by distillation with isothermal holding at 380 °C.

In [14], shale SCR was produced from combustible shale at T = 560 °C, then distilled up to 410 °C, yielding 29.3% pitch with T_s = 55.5 °C, α_1-fraction content is ~0.61%, and volatile matter yield is 83.7%. In [15], shale SCT distillation up to 360 °C produced pitch with 59% yield.

When distilling peat SCT (obtained by pyrolysis at 600 °C) up to 340 °C, pitch with yields 18-24% were obtained with phenol content of 6% [16]. In [17] on peat SCT distillation was shown: light fraction yield (up to 170 °C) is 5.3%; fraction yield at T = 170-200 °C is 11.2%; fraction yield at T = 200-230 °C is 12.1%, with maximum narrow fraction yield of 26.7% observed at T = 230-270 °C, and pitch yield is 24.3%. Study [18] established that 4.8% phenols remained in the pitch during peat SCT distillation.

Indeed, SCT production as raw material for pitches mainly occurs in a reducing environment (native gas atmosphere). Of particular interest is the production of pitches from SCT as a liquid pyrolysis product in an inert atmosphere. Also noteworthy is the comparison of pitch characteristics depending on the production environment of the source material.

2. Production of pitch from SCT (semi-coking tar) as a liquid product of coal pyrolysis in inert and reducing atmospheres [19, 20].

This section describes the experimental equipment for producing semicokes and SCT from coals. The methodology for pitch extraction from SCT is also presented. The described SCT production techniques were previously developed and validated by our research team in 19-20]. This paper provides their brief overview.

2.1 Methods of SCT extraction from coals. Fig. 1 shows the unit for SCT extraction from coals in an inert atmosphere. Fig. 2 shows the unit for SCT production in a reducing atmosphere.

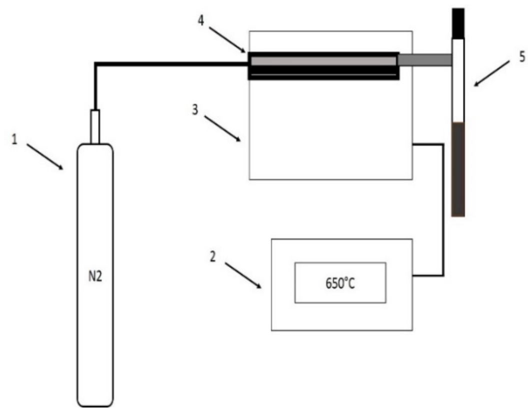

Fig. 1 Apparatus for semi-coking in an inert atmosphere:
1 – gas cylinder; 2 – control unit; 3 – Gray-King assay apparatus body; 4 – quartz retort; 5 – tar collector [19].

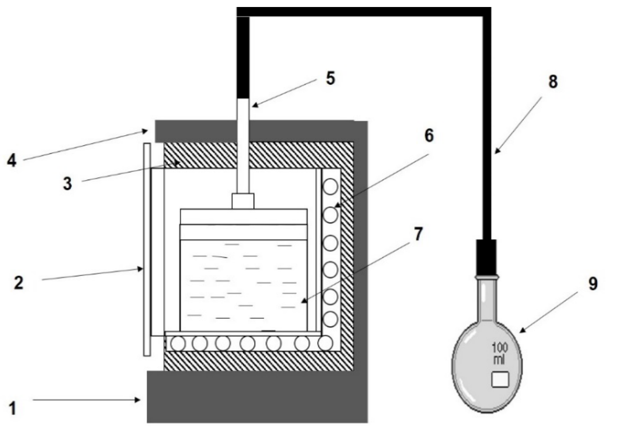

Fig. 2 Apparatus for semi-coking in a reducing atmosphere (side view):
1 – furnace base with integrated control system; 2 – furnace door; 3 – thermal insulation material; 4 – muffle furnace body; 5 – furnace gas outlet pipe; 6 – electric heating system of the furnace; 7 – coking box with coal, covered by a lid with a round opening; 8 – pipeline for transporting vapor-gas products; 9 – tar collector (flask-type) [20].

The apparatus for SCT production (Fig. 1) in an inert atmosphere consisted of a gas cylinder (1) and a Gray-King assay apparatus. The pyrolysis was conducted in the assay apparatus under a nitrogen atmosphere. The gas cylinder was connected via silicone tubing to a quartz retort containing the coal sample (4). The control unit regulated the electric heating system was integrated into the apparatus housing (3). Pyrolysis was performed at 650 °C, with semi-coking liquid products (SCT) collected in a glass test tube (5).

The SCT production apparatus (Fig. 2) for reducing conditions comprised: an EPLK-2 muffle furnace for coal carbonization (4); an SCT collection system consisting of: built-in exhaust gas piping (5); silicone transfer lines for vapor-phase products (8); glass flask condensers (9) for liquid product recovery.

2.2 Methodology for pitch extraction from SCT. The primary approach for pitch extraction was implemented for fundamental distillation scheme of tar processing. It is shown in Fig. 3.

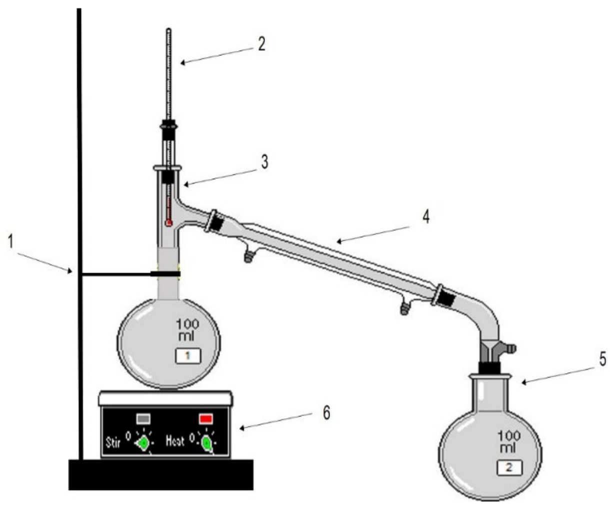

Fig. 3. Schematic diagram of pitch extraction by tar distillation [20]:
1 – stand; 2 – thermometer; 3 – flask containing tar; 4 – condenser; 5 – distillate collector; 6 – flask heater.

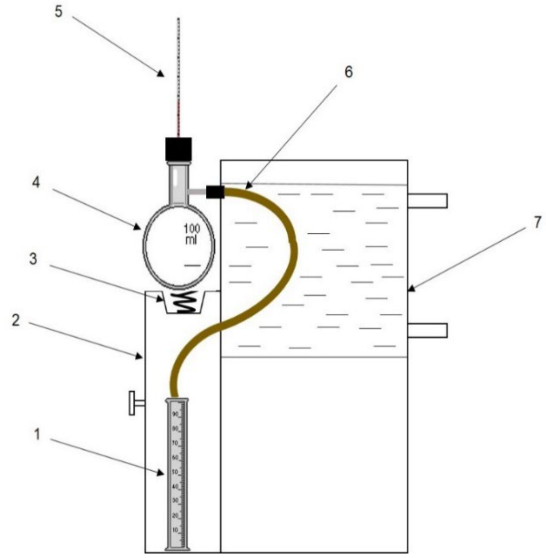

Fig. 4. Schematic of the SCT fractional distillation apparatus using ARN-LAB 03 device (side view) [20]:
1 – receiver flask; 2 – control unit; 3 – built-in flask heater; 4 – Wurtz flask; 5 – thermometer with fixation stopper; 6 – distillate outlet valve;
7 – condenser.

Pitch extraction is achieved by heating the tar to separate its fractions into gaseous components. They are then cooled in the condenser (4) to liquid state and collected in the receiver (5) [21].

In our previous studies [19, 21–22], pitch extraction from SCT through fractional distillation was performed using the ARN-Lab-03 apparatus (Fig. 4). The apparatus for SCT-derived pitch isolation consisted of: a control unit with heating adjustment buttons and a flask positioning knob, a built-in flask heater (3), a condenser (7) supplied with cooling water via an integrated tubing system.

Table 1 presents the fractional distillation results of SCT obtained from Zh and GZh coal grades in an inert atmosphere [19, 21].

Table 1. Yields of fractions of coal grades Zh and GZh [19, 21]

T, °C	Yield of coal grade Zh tar fractions, %	Yield of coal grade GZh tar fractions, %
30 – 99	31.0	13.1
99 – 130	24.4	51.0
130 – 360	33.3	16.2
> 360	11.3	19.7
Losses + gases	—	1.1

The first distillate droplet appeared at T = 86 °C for SCT derived from GZh and Zh coal grades. Table 1 demonstrates that the highest pitch yield was observed for GZh coal SCT. Key conclusions from Table 1 include:

1. The maximum fraction yield (60-70%) for these tars corresponds to the light fraction of coal tar [19, 21].

2. For Zh coal tar was found, that 31% of its content distilled below 99 °C [19, 21].

These findings highlight the potential for solvent-based pitch extraction from such SCTs. For instance, reference [23] discusses toluene-assisted pitch extraction from SCT. In our study [20], the extraction process was conducted using a modified ARN-Lab-03 apparatus (Fig. 4), where the Wurtz flask was charged with SCT and toluene. Distillation continued up to toluene's boiling point (110 °C).

Table 2 presents previously published data [20] on pitch extraction from SCT obtained under reducing atmosphere from GZh and K coal grades.

Table 2. Yield of pitches extracted from SCT by toluene-assisted extraction [20]

№	Name	SCT-derived pitch yield, %
1	SCT GZh + toluene (40/80)	20.2
2	SCT GZh + toluene (40/100)	12.0
3	SCT GZh + toluene (40/12)	5.0

Table 2 shows, that the pitch yield decreased for GZh SCTwith increasing solvent volume, while for K SCT the pitch yield was 5% [20].

This work presents the results of determining the extraction time of pitches from tars obtained in a reducing atmosphere in reference [20]. Table 3 shows the duration of heating SCT from room temperature to complete recovery of fractions (t) at 110 °C and the duration τ from the appearance of the first droplet to complete recovery of fractions at 110 °C. Δt is the preheating duration of the tar before the extraction process ($\Delta t = t - \tau$).

Table 3. Heating time of SCT and extraction period.

№	Name	t, min	τ, min	Δt, min
1	SCT GZh + toluene (40/80)	180	90	90
2	SCT GZh + toluene (40/100)	170	90	80
3	SCT GZh + toluene (40/12)	165	80	85

According to Table 3, the tar preheating duration (Δt) at all SCT/toluene ratios until first droplet appearance ranged between 80-90 minutes. With increasing toluene volume Δt decreased for GZh SCT from 90 to 80 minutes. For K SCT Δt remained constant at 85 minutes. The extraction duration τ showed no variation (τ = 90 minutes) with increasing toluene volume for GZh SCT.

3. Characteristics of pitches extracted from SCT. This section presents the data and characteristics of the pitches we previously obtained from SCT. In this work, we determined the softening temperature (T_{melt}) of the pitches. The T_{melt} determination process proceeded as follows: portions of pitch were separated from different surface areas of the obtained pitch. The separated portions were heated, and the melting process was monitored using a microscope. The initial melting temperature was determined by a secondary instrument connected to the heating element with a thermocouple. The transition of pitch to liquid state was considered as melting. Additionally, for the finished pitch from GZh SCT (tar + toluene (40/80)), we conducted a low-temperature carbonization experiment by heating to 550°C and holding for 1 hour. The carbonization was conducted in a muffle furnace, where pitch samples were placed in ceramic cups and covered with a metal lid. We determined the carbonizate yield K_{550} as the percentage ratio of pitch mass to the obtained carbonizate mass. For the carbonizate, we determined the volatile matter yield $V^{(daf)}$ according to GOST R 55660-2013.

3.1 Composition and properties of pitches from SCT obtained in an inert atmosphere. Table 4 presents the characteristics of pitch derived from Zh-grade SCT.

Table 4. Characteristics of pitch obtained from Zh-grade coal SCT [19].

Name	T_S, °C	γ, %	β, %	α, %	$α_2$, %	$α_1$, %	X, %	Ash content, %
Pitch (Zh-grade coal SCT)	51	30.4	65.5	4.1	3.6	0.5	57.5	0.17

The melting temperature T_{melt} at which the pitch transitioned to liquid state was 55 °C for pitch from Zh SCT. For pitch from GZh SCT, the melting onset occurred at $T_{melt} \approx 68$ °C and $T_{melt} \approx 210$ °C for different pitch samples, indicating potential heterogeneity of the product.

In this study, extraction from Zh SCT using toluene (at a pitch/toluene ratio of 1/1, up to toluene's boiling point of 110 °C) produced pitch with melting point T_s = 56 °C; pitch yield = 10%; volatile matter content = 79.4%, and ash content = 0.98%. For this pitch, T_{melt} varied between 62-81 °C across different sample areas.

3.2 Composition and properties of pitches from SCT obtained in reducing atmosphere.

Table 5 shows the fractional composition of extracted pitches from SCT produced under reducing conditions from GZh and K coals reducing atmosphere [20].

Table 5. Fractional composition of pitches derived from SCT of GZh and K coal grades [20].

№	Name	T_S, °C	*γ*, %	*β*, %	*α*, %	$α_1$, %	X, %	Ash content, %
1	Pitch (GZh-grade coal SCT)	36	33.8	44.7	11.5	0.5	72.6	0.04
2	Pitch (K-grade coal SCT)	46	-	-	13.4	0.3	67.5	0.07

According to Table 5, the pitches from SCT produced in a reducing atmosphere are soft based on their T_s values. The pitches also have low content of *α* and $α_1$-fractions compared to commercial pitches according to [20]. For the pitch (GZh SCT), T_{melt} is about 49 °C, while for the pitch (K SCT), $T_{melt} \approx 48$ °C.

3.3 Low-temperature carbonization of pitches. This section presents an experiment on low-temperature carbonization of pitch obtained from GZh SCT (pitch data shown in Table 4) and commercial electrode pitch grade B (*α* = 25.8%, $α_1$ = 4.5%). The characteristics of this pitch are described in [20]. The carbonization was performed by heating at a rate of 7.5 °C/min with a 1-hour holding time. Table 6 presents the carbonizate data.

Table 6. Yield of pitch carbonizates and technical analysis data.

№	Name	K_{550}, %	$V^{(daf)}$, %
1	K-grade coal (SCT)	34.4	50.0
2	K(B)	59.1	10.0

Table 6 shows that the K(SCT) yield is lower compared to K(B). Also, the volatile matter yield of carbonizate K(SCT) was 50%.

Discussion and conclusion

The pitch obtained by distillation of SCT (as a liquid pyrolysis product of Zh-grade coal in an inert atmosphere) showed a softening temperature T_s = 51 °C. It was higher than those of pitches produced by extraction from SCT (as pyrolysis products of GZh and K coals in a reducing atmosphere). The pitch produced by distillation of SCT (as a pyrolysis product of GZh-grade coal in an inert atmosphere) exhibited significant variations in melting temperature across different sample regions, indicating product heterogeneity. This phenomenon might be a result of the need for lowering the final distillation temperature for this particular tar. At temperatures exceeding 300 °C, the distillation process continued, potentially causing simultaneous evaporation of light pitch fractions (γ- and β-fractions) with the distillates, ultimately leading to non-uniform product composition.

The carbonizate yield K_{550} was higher for grade B electrode pitch than for pitch from SCT. This difference might be a result of the higher α_1-content in grade B pitch compared to SCT-based pitch. The α_1-fraction content in pitch contributes to increased carbonizate yield, as demonstrated in [25-26]. Furthermore, pitches obtained from SCT through distillation showed lower α- and α_1-fraction contents compared to pitches produced by distillation of industrial coal tar in [26].

The extracted and distilled pitches from SPK Zh (the tar was obtained in an inert environment) in this work were soft. The extracted pitch showed higher volatile matter content (X) compared to pitch obtained by distillation. The ash content was also higher for the extracted pitch relative to the distilled pitch. The melting temperatures T_{melt} exceeded the temperatures T_s for all pitch samples. Furthermore, pitches obtained by extraction from SCT (as liquid semi-coking products of GZh and K coals in reducing atmosphere) demonstrated higher α-fraction content and lower ash content, when compared to pitch produced by distillation from SCT (as liquid semi-coking product of Zh coal in inert atmosphere).

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