

ANALYSIS AND SELECTION OF COMPOSITE RAW MATERIALS
FOR CARBON BLACK PRODUCTION*Dmytro Sheremeta¹, Vasyl Bohun¹, Kateryna Roienko¹,
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Abstract. The availability and possibility of using various alternative types of raw materials for carbon black production have been analyzed. The quality of raw materials was evaluated according to the aromaticity value, correlation index, carbon content, and hydrogen content. Composite mixtures were created and tested under industrial conditions. Two programs for calculating the theoretical yield of carbon black by balance and thermos-chemical methods have been developed and tested. An evaluation of the carbon black cost was carried out under the conditions of using the residue of tire processing – pyrolysis oil – as a fuel.

Keywords: carbon black, composite raw material, correlation index, yield of carbon black, program for calculation, prime cost.

1. Introduction

Carbon black is a very finely dispersed powder-like elemental product consisting of fused aggregates and almost spherical particles.¹ It typically contains more than 97% of carbon with some sulfur, oxygen, and hydrogen.² Carbon black products are classified by the size of the primary particles, their aggregation, and the presence of certain impurities. Aggregates, which it quickly forms, represent a chain of hundreds of primary spherical particles with a size of 100 to 800 nm and above, and merged into a randomly developed structure.³

Recently, there has been an increase in the areas of application of carbon black. In addition to its traditional use in the tire industry (almost 70% of the produced carbon black is aimed at increasing the strength of rubber and increasing its resistance to abrasion), it is actively used in the production of alloys, as a black pigment in paint and coatings, because it intensively absorbs ultraviolet radiation, which leads to degradation of metals,⁴ in the polymer and electrochemical industries, special copying types of paper and in the production processes of ribbons for printers, thanks to the high strength and stability of coloring.

The application of carbon black also extends to the fields of electronics, fuel elements,⁵ hydrogen storage,⁶ lithium-ion batteries,⁷ chemical sensors, biosensors,⁸ nanoscale electronic devices, and supercapacitors, as a conductor of electricity.⁹ In addition, carbon black particles are used in radio-absorbing materials, as they reduce the cross-section of aircraft radar.^{10,11}

Alternative methods for carbon black production are proposed, which are based on the use of renewable resources, such as algae and plant waste from agriculture.^{12–14} The process includes hydrolysis, carbonization, and pyrolysis of green algae and sugar cane. Despite the high carbon content (about 90% in the product), an extremely high specific surface area of 605 m²/g, this three-stage technology attracts toxic reagents (concentrated sulfuric acid), and in large quantities (the ratio is 1:30 g/mL), dictates the need to carry out secondary pyrolysis (the temperature of the process should reach 900°C), too high cost factors, weak economy in comparison with commercial proven technology, almost nullify the prospect of carbon black production from this type of alternative plant raw material.

The use of rice husk^{15,16} for carbon black production involves plasma technology and the operation of an aerosol reactor,¹⁷ which, in principle, would make it possible to abandon non-renewable petroleum raw materials. But the logistics of supplying plant raw materials (Egypt, Abu Dhabi, China), the weak possibility of growing algae in

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large volumes, their collection, and storage in the conditions of Ukraine make it almost impossible to develop this direction of creating a raw material base.

As a result of the search for raw materials of free carbon black in a non-commercial volume, juice production waste, namely orange peel, was found as a highly effective absorbing material of heavy metals from wastewater.¹⁸ In this case, they were used as a source for obtaining a new microporous nanoactivated carbon using the method of chemical activation of zinc chloride.

A promising method of disposing used car tires by pyrolysis with carbon black production^{19,20} deserves attention. The product has too high percentage of ash content, and for practical use, it must be enriched. Simultaneously formed liquid fuel and pyrolysis gas can be used for boilers and furnaces, where the destruction of the tire material will take place directly.

In addition to the ecological aspect of this direction of selecting raw materials for carbon black production, it is necessary to evaluate the economic opportunities and energy potential during the processing of used car tires. It is important to take into account the experience gained by other countries in the field of tire recycling and integrate different methods in our country.

Up-to-date, carbon black production remains one of the branches of petrochemicals, which is related to the processing of raw materials of petroleum origin and products of coke production. Recently, significant changes have been taking place in carbon black production, related to the expansion of the raw material base, namely the creation of starting mixtures that include components with sufficiently different properties and indicators.

Liquid co-products of the petrochemical or coke industry are mostly used as raw materials for carbon black production. The list of components that can be used includes a wide variety of aromatized products – thermal gas oil, thermal resin, thermal oil, catalytic cracking and coking gas oils, extracts of selective oil purification, secondary gas oils, pyrolysis distillates and resins, low-temperature coal tars, anthracene fraction, pitch distillate, *etc.*

There is a relationship between the correlation index and the dispersion of carbon black (the specific surface area of its particles): the higher the correlation index, the higher the dispersion (S_{pyt}) of carbon black - from 40 m²/g (for carbon black N550) to 115 m²/g (N220), therefore, to obtain highly dispersed grades of carbon black (S_{pyt} > 100 m²/g), it is necessary to use raw materials with a correlation index of 125 – 130. For the production of low-activity grades of carbon black, it is possible to use petroleum raw materials with a correlation index of 90. The raw materials, as a rule, are fractions with a molecular weight of 150 – 400 units and a boiling point of 170 – 500°C. The density of

various types of raw materials and their mixtures ranges from 0.84 to 1.14 g/cm³. The density of the raw material provides valuable primary information about its quality. The higher the density of the raw material, the more condensed aromatic hydrocarbons it contains, the higher the yield of carbon black from this type of raw material will be. So, for example, the anthracene fraction with a density of 1.089 g/cm³ contains more than 20% of naphthalene (bicyclic) hydrocarbons, while the fraction with a density of 1.12 g/cm³ contains practically none, but contains much more polyaromatic hydrocarbons.

The viscosity, like the density, and average volumetric boiling temperature indirectly characterize the quality of raw materials.²¹ All these indirect properties are based on differences in the properties of different classes of hydrocarbons. Heavy paraffinic hydrocarbons and pitches are characterized by increased viscosity. The high viscosity of the raw material indicates a significant content of these classes of hydrocarbons.

The dependence of the viscosity of the raw material on the content of aromatic hydrocarbons in it can be used in combination with other properties of hydrocarbons to assess its suitability for obtaining carbon black.

The composition of raw materials is not stable in terms of chemical components and may vary even from one supplier. In addition, the limitation of the traditional raw material base, developed from the point of view of technology, especially as a result of military operations and the shutdown of several coke-chemical enterprises, leads to the need to expand it by attracting new components. This raises the question of creating a universal tool for determining the optimal composition of raw materials for each lot, with the ability to vary the hydrocarbon composition when using coproducts of petroleum and coke production.

An attempt was made to investigate the properties and influence of the created hydrocarbon compositions on the quantity and quality of the finished product, which would make it possible to improve and expand the raw material base of the country in difficult modern conditions. The goal was to select acceptable optimal compositions that could replace the missing traditional raw materials in the production of high-quality domestic carbon black.

2. Experimental

2.1. Materials

For the calculations and selection of the compositions of the mixture, we used the production data of technical carbon (Table 1).

Table 1. Types of produced raw materials

Name of analysis	Petroleum types of raw materials					Coke-chemical types of raw materials			
	Fuel oil M100	Tar oil	HCG	HCG*	Pyrolysis oil	Anthracene	DKHZ	AMKHZ	AKHZ
Analytical moisture, W ^a , %	0.00	0.00	0.00	0.0	0.00	0.00	0.86	2.04	10.64
Analytical ash, A ^a , %	0.00	0.023	0.051	0.0	0.00	0.00	0.05	0.09	0.08
Carbon, C ^{daf}	84.56	88.54	88.92	83.67	90.95	91.00	93.24	91.90	93.64
Hydrogen, H ^{daf}	11.44	10.13	9.05	8.24	5.83	5.95	4.87	4.49	4.28
Sulfur, S ^d , %	0.96	1.07	0.69	0.11	0.24	1.11	1.63	0.86	1.38
Nitrogen+oxygen, (N+O) ^{daf} , %	3.54	0.26	1.34	7.98	3.01	1.94	0.26	2.75	0.70
C ^{daf} /H ^{daf} , units	7.39	8.74	9.83	10.15	15.60	15.29	19.15	20.47	21.88
H/C, units	1.62	1.37	1.22	1.18	0.77	0.78	0.63	0.59	0.55
Aromatic value, δ, units	2.89	4.64	5.86	6.24	9.53	9.35	10.69	11.01	11.37
Correlation index	72.2	102	114	127	144	160	184	188	206
Density at 20°C, g/cm ³ , min	0.9-1.1	1-1.25	1.00		1.0	1.12			

DKHZ – Kametstal Coke Chemical Plant, AMKHZ – Coke production ArcelorMittal Kryvyi Rih, AKHZ – Avdiivka Coke Chemical Plant, HCG* – MOL (Hungary)

2.2. Methods

To assess the degree of aromatization of raw materials, an empirical indicator is used – the correlation index (CI):

$$CI = 437 \cdot \rho_{20} - 456,8 + 48940/T_{boil} \quad (1)$$

where ρ_{20} is a density of raw materials at the temperature of 20 °C, g/cm³; T_{boil} is the temperature of the average boiling volume of hydrocarbons, K.

To evaluate the effectiveness of the formulations of the created mixtures, tests were conducted under production conditions to check the quality of the obtained carbon black.

To predict the quantitative yield of the target product depending on the content of carbon and hydrogen in the raw materials, and their subsequent correction, a software product was developed and created using two calculation methods.

The first method is based on the material balance equation for obtaining carbon and includes the determination of the mass of carbon in raw materials, nitrogen, and oxygen in waste gases.

$$G_{cb} = G_{raw\ mat.} \cdot \frac{\%C_c}{100} + G_{nat.gas/carbon} - G_{was.gas/carbon} \quad (2)$$

are masses of carbon in natural gas and waste gases, kg/h; %C_c is a carbon content in raw materials, wt %. The mass of carbon in natural gas was calculated according to the formula:

$$G_{nat.gas/carbon} = Q_{nat.gas} \cdot 0.7 \cdot \frac{75}{100} = Q_{nat.gas} \cdot 0.525, \quad (3)$$

where $Q_{nat.gas}$ is a consumption of natural gas, nm³/h; 0.7 is a density of natural gas, kg/nm³; 75 is a carbon content in natural gas, wt %.

Thus, the mass of carbon in natural gas is:

$$G_{nat.gas/carbon} = Q_{nat.gas} \cdot 0.525 \quad (4)$$

The carbon content in waste gases was determined by gas analysis. The composition of the gases was calculated for each component under the assumption that oxygen is not present in the gases:

$$\%CO_2^i = \%CO_2 \cdot \frac{100}{100 - \%O_2} \quad (5)$$

$$\%H_2^i = \%H_2 \cdot \frac{100}{100 - \%O_2}, \quad (6)$$

Accordingly, the nitrogen content was:

$$N_2^i = 100 - (CO_2^i + CO^i + H_2^i + 0.7), \quad (7)$$

where 0.7 is a content in waste gases of SO₂, H₂S, CH₄.

The volume of waste gases:

$$Q_{was.gas} = Q_{air} \cdot \frac{79}{N_{2was.gas}}, \quad (8)$$

where Q_{air} is a flow rate of air entering the reactor, nm³/h; 79 is a nitrogen content in the air, % vol.; $N_{2was.gas}$ is a nitrogen content in waste gases, % vol.

$$G_{carbon}^{was.gas} = Q_{was.gas} \cdot \left(\frac{CO + CO_2}{100} \right) \cdot \frac{12}{22.4}, \quad (9)$$

The yield of carbon black is:

$$B_{cb} = \frac{G_{cb}}{G_{raw\ mat.}} \cdot 100\% \quad (10)$$

The second calculation method was based on the dependence of carbon black yield on fuel and air consumption and included thermochemical calculations of the incomplete combustion process.

$$B = b - bFV_{air} + bHg_{fuel}, \quad (11)$$

where B is a carbon yield, kg/kg of raw material; b is a carbon yield coefficient, equal to the carbon content in the raw material or the carbon content in the cyclic structures of the raw material molecules; F , H are coefficients determined by the mass fraction of carbon and hydrogen in raw materials and fuel; V_{air} is an air consumption, m³/kg of raw material; g_{fuel} is fuel consumption, kg/kg of raw material.

$$F = \frac{0.21}{1.30C_{raw\ mat.} + 4.13H_{raw\ mat.}}, \quad (12)$$

$$H = \frac{1.30C_{fuel} + 4.13H_{fuel}}{1.30C_{fuel} + 4.13H_{fuel}}, \quad (13)$$

C_{fuel} , $C_{raw\ mat.}$ are carbon content in fuel and raw material, respectively, kg/kg; H_{fuel} , $H_{raw\ mat.}$ are hydrogen content in fuel and raw material, respectively, kg/kg.

The last method included two options for fuel: the first option is the use of natural gas (method 2.1), the second option (method 2.2) is the combustion products of pyrolysis oil (a heavy, tarry product of tire processing).

An application based on the Python programming language was developed, which made it possible to calculate the yield of carbon black according to the presented methods.

3. Results and Discussion

Five types of alternative raw material mixtures were studied and compared with the traditional raw material, the anthracene fraction obtained by the rectification of coal tar. The optimal selection of the composition of raw mixtures was carried out based on the results of the developed application. Visualization of the calculation results of the program for two-component mixture No. 2 and three-component mixture No. 3 is presented in Fig. 1.

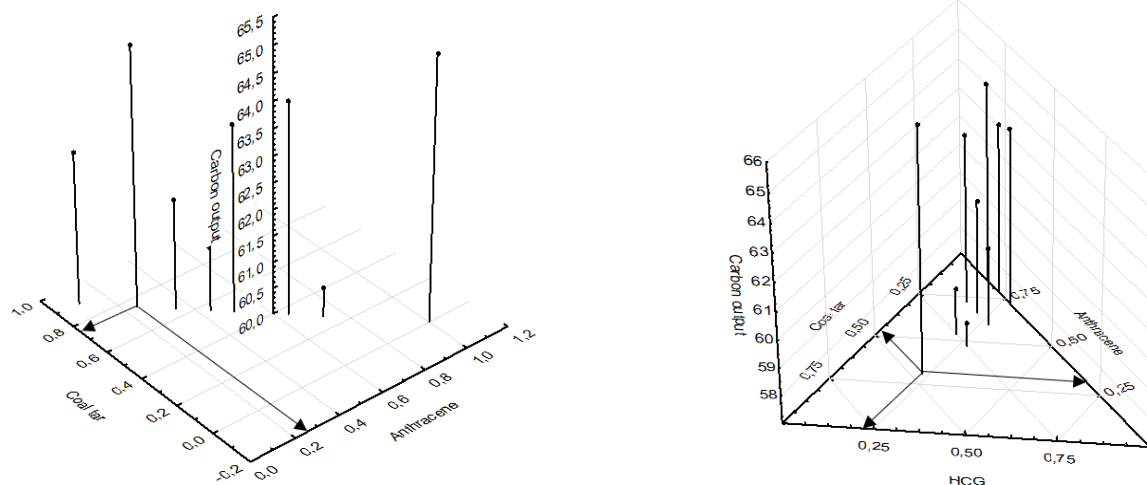


Fig. 1. Visualization of the results of the calculation of raw material mixtures No. 2 (anthracene – 25%, coal tar – 75%) and No. 3 (anthracene – 31%, coal tar – 46%, HCG – 23%)

The result of theoretical calculations and their comparison with the practical carbon black yield is presented in Fig. 2. The numbering of mixtures according to the composition and characteristics is given in Table 2.

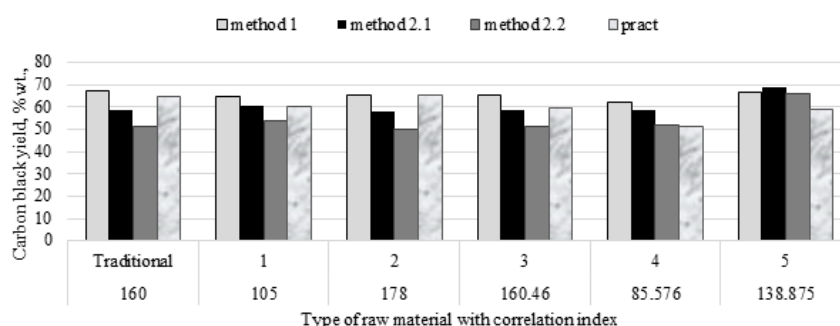
It can be seen from the graphic chart that the carbon black yield, calculated by various methods, is slightly different. The reason for this may be the influence of the type, quantity, and quality of fuel on the process of carbon

black production. If we compare the theoretical yield with the practical data, the similarity of the results is observed, the existing deviation may be the reason for the fluctuation of the temperature regime, the inaccuracy of the determination of the carbon content in the raw material and the composition of the waste gases, namely the hydrogen content.

The determined properties of the selected mixtures are shown in Table 2.

Table 2. Characteristics of traditional raw materials and created mixtures for carbon black production

Raw material		Parameters						
		Density, g/cm ³	Viscosity for 50°C, mm ² /s	Carbon content C, % wt.	Hydrogen content H, % wt.	C/H value	Aromaticity value	Correlation index
Traditional raw material								
Anthracene fraction	100%	1.120	10	91	5.2	16.84	9.35	160
Mixture No.1								
Heavy catalytic gas oil (HCG)	25%	0.997	281.25	88.63	9.86	9.01	4.95	105
Tar oil	75%							
Mixture No.2								
Anthracene	25%	1.172	85	89.65	5.5	16.15	8.34	178
Coal tar	75%							
Mixture No.3								
Anthracene	31%	1.129	57.15	89.69	6.27	14.80	7.93	160.46
Coal tar	46%							
HCG	23%							
Mixture No.4								
HCG	32%	1.040	256.4	85.96	10.68	8.05	3.84	85.58
Fuel oil M100	68%							
Mixture No.5								
Pyrolysis oil	85%	1.073	—	90.63	6.36	14.25	7.05	138.88
Darkened fraction	15%							

**Fig. 2.** Carbon black yield depends on the type of raw material of the process

Using various mixed compositions as raw materials, it is possible to observe the influence of the type of raw materials on the yield of the finished product, *i.e.*, in the process of involving a mixture containing heavy residues of oil refining, the carbon black yield decreases. This can be explained by the low aromaticity value (3.84) and correlation index (85.58) of the created mixture No. 4. The carbon black yield from raw materials when using fuel oil

is slightly reduced (9%), however, giving the relatively low cost of fuel oil compared to pyrolysis oil, its use can be economically justified even at the given level of existing technology.

Based on calculation data and balance tests, raw materials No. 2 (a mixture of anthracene fraction and coal tar) and No. 3 (a mixture of anthracene fraction, coal tar, and heavy catalytic gas oil) perform best of all. These

mixtures have the highest correlation indices and aromaticity value.

Simultaneously with the calculation of carbon black yield based on the determined component composition of raw material mixtures, some physical-chemical quality parameters of carbon black were analyzed under industrial conditions and compared with the values from the specification for carbon black N550.

Determinations were carried out according to standard methods, the numbering of which is presented with the results of analyses in Table 3.

Due to the optimization of the raw material composition, which took into account the quality characteristics of the components, we managed to obtain products with almost all physico-chemical parameters within the normalized values.

Table 3. Quality indicators of technical carbon samples obtained from different types of mixed raw materials

Iodine Adsorption Number, g/kg	Traditional raw materials (anthracene fraction)	Raw material mixture					Norm	Method
		Mixture No.1	Mixture No.2	Mixture No.3	Mixture No.4	Mixture No.5		
Iodine adsorption number, g/kg	42.8	38.7	44.6	42.9	35.0	44.2	38.0-48.0	ASTM D 1510-21
Oil absorption number, cm ³ /100g	121.1	114.5	120.3	122.4	106.4	119.5	116.0-126.0	ASTM D 2414-23
Oil absorption number, Compressed Sample, cm ³ /100g	81.7	76.8	83.9	82.7	73.5	81.8	85.0±7.0	ASTM D 3493-21
External Surface Area by Nitrogen Adsorption (STSA), m ² /g	36.1	36.8	40.6	37.1	38.2	39.3	39.0±6.0	ASTM D 6556-21
Total surface area by nitrogen adsorption (NSA), m ² /g	38.3	37.9	41.1	38.9	40.1	41.2	40.0±6.0	ASTM D 6556-21
Transmittance of toluene extract, %, min	97	85	92	98	78	96	85	ASTM D 1618-18
Automated individual pellet hardness, g	33.3	38.9	38.6	33.4	42.4	37.9	10.0-50.0	ASTM D 5230-21
Sieve residue, 45 µm (325 USStd Sieve), ppm,	37	307	834	469	250	247	Max 1000	ASTM D 1514-23
pH value	9.1	8.0	9.4	8.1	8.1	9.4	7.0-10.0	ASTM D 1512-21
Fines content, %,	0.8	1.0	0.8	1.0	0.8	1.5	Max 15	ASTM D 1508-12 (2017)
Moisture content, % wt.	0.45	0.52	0.59	0.51	0.56	0.54	Max 1.0	ASTM D 1509-18
The fraction of surface area of micropores, %	5.74	2.90	1.22	4.63	4.74	4.61		
The degree of branching of the secondary structure of carbon black, %	48.23	49.09	43.38	48.00	44.76	46.09		

The influence of the correlation index on the structure of carbon black cannot be presented in the form of any tables or graphs without taking into account other properties of the raw material. The technique of increasing oil absorption by increasing the content of coke chemical raw materials in the raw material mixture is used and justified, while increasing the degree of aromatization of catalytic gas oil with an index of 114 points (Table 1) by adding pyrolysis resin with a correlation index of 144 (Table 1) does not lead to an increase in the structure of carbon black. This may be related to the presence of a large amount of asphaltene in the pitch, which adversely affect

the structure of the formed carbon black. The presence of heterocyclic compounds in the raw material also leads to a decrease in the structure of carbon black, which is evidenced by a decrease in the oil absorption index to 106.4 (mixture No.4), 114.5 (mixture No.1, 119.5 (mixture No.5), and as a result – reducing the size of carbon black aggregates due to the separation of its particles.

The increased value of the transmittance coefficient of the toluene extract in almost all mixtures (for example, 97.6 – mixture No. 3) indicates a small amount of condensed raw materials on the surface of carbon black particles, which indicates that the process does not require

a temperature correction in the reaction zone of the reactor. Usually, increasing the temperature in the reactor to reduce the toluene extract rate can lead to a decrease in the biocarbon yield.

Carbon black obtained from raw materials No. 1, 4, 5 is characterized by a lower level of secondary structure (OAN) than in the comparison standard, and OAN in lots from raw materials No. 2, 3 is within the normalized values (Table 3).

Fluctuations in the level of physical-chemical characteristics between individual samples of carbon black did not exceed the normalized limits. (Table 3). The fraction of the surface area of pores with a diameter of less than 2 nm M_p (%), calculated according to formula (1), in carbon black obtained from raw material mixture No. 4 is about 6% of the surface area of all pores, in carbon black from mixture No. 1 – about 3 %.

$$M_p = \frac{NSA - STSA}{NSA} \cdot 100, \quad (14)$$

where M_p – micropore area fraction; NSA – total specific surface area of carbon black by low-temperature nitrogen adsorption; $STSA$ – external specific surface area by the nitrogen statistical layer.

The structural strength of rubber is determined by the ability of carbon black to form secondary structures in rubber. The degree of branching of the secondary structure of carbon black was calculated using the formula:

$$D = \frac{OAN - COAN}{OAN} \cdot 100, \quad (15)$$

where D – the degree of branching of carbon black secondary structure; OAN – oil absorption; $COAN$ – oil absorption of compressed sample.

Therefore, we can expect a different distribution of molecular masses of rubber in the matrix and interphase layer of the compared options.

According to experimental data, the degree of branching of carbon black secondary structure from mixture No. 1 is higher than that of the product from raw material No. 5 and to a greater extent than that of carbon from mixture No. 2 (Table 3).

The quality of raw materials significantly affects the technical and economic indicators of production, especially the carbon black yield, equipment capacity, specific consumption of electricity, and water.

To assess the possible use of raw material mixtures, the prime cost of carbon black was calculated under the conditions of using the residue of tire processing, pyrolysis oil,²² as a fuel.

The calculation results are shown in Table 4.

The indicators are significantly underestimated, since the value of “non-standard” secondary products, which are now sold by individual entrepreneurs, was used in the calculation. If, for example, high-quality pyrolysis carbon – analogue of N772/N550/N660 is sold to plants of

rubber engineering products as an additive, then its price can reach UAH 2.000/t and the results will be significantly higher. The presented data show that the involvement of the proposed components in the process of carbon black production is profitable from an economic point of view.

Table 4. Calculation of the prime cost of the product

№	Name	Cost
1	Carbon black production, thousand tons/year	19.9147248
2	Semi-variable costs:	
	raw material:	23.9292
	tar, thousand tons/year	4.2228
	darkened fraction, thousand tons/year	16000
	tar, UAH/t	10000
	tar oil, UAH/t	26000
	heavy catalytic gas oil, UAH/t	14000
	fuel, UAH/t	342148572
	The cost of raw materials for the entire production, UAH	9081
	Additional materials, UAH/t	180845616
	Total for products, UAH	80450450.5
3	Semi-fixed costs, UAH	603444638
4	Total cost, UAH	30301.4299
5	Prime cost of products, UAH/t	47679.3
6	Sales price of products (without VAT), UAH/t	949520139
7	Revenues from sales, UAH/year	346075500
8	Profit, UAH/year	

4. Conclusions

The option of creating raw material mixtures involved in the production process of carbon black co-products of oil refining and coke chemistry is acceptable as the main solution to the problem of expanding the raw material base. The deviation (within 1.5-3%) of the calculated values according to the developed algorithm from the practical carbon black yield, with the preservation of quality indicators within the limits of the standards according to the presented recipes, confirmed the perspective of using the developed methods of estimating the carbon black yield. The possibility of forecasting during the development of effective formulations of raw materials with the simultaneous solution of disposal methods of heavy co-products of oil, coal and used tire processing processes was revealed. The direction of using waste tires is considered a promising option, both from the decarbonization of the industrial carbon black production and from the economic point of view.

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АНАЛІЗ І ПІДБІР КОМПОЗИЦІЙНОЇ СИРОВИНИ ДЛЯ ВИРОБНИЦТВА ТЕХНІЧНОГО ВУГЛЕЦЮ

Анотація. Проаналізовано наявність і можливість використання різноманітних альтернативних видів сировини для виробництва технічного вуглецю. Здійснено оцінювання якості сировинних матеріалів за показником ароматичності, за індексом кореляції, вмістом вуглецю та водню. Створено й апробовано в промислових умовах композиційні суміші. Розроблено та перевірено дві програми розрахунку теоретичного виходу технічного вуглецю за балансовим і термохімічним методами. Проведено оцінювання собівартості технічного вуглецю в умовах використання як палива залишки переробки шин – піролізної оливи.

Ключові слова: технічний вуглець, композиційна сировина, індекс кореляції, вихід технічного вуглецю, програма розрахунку, собівартість.