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UTILIZATION OF THE PREPYROLYZED TECHNICAL HYDROLYSIS LIGNIN AS A FUEL FOR IRON ORE SINTERING

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Перспективним напрямком утилізації технічного гідролізного лігніну є його застосування в металургійному виробництві, в першу чергу при підготовці залізорудної сировини і доменному процесі. Значний резерв при цьому зосереджений в агломераційному процесі. Для поліпшення паливних властивостей лігніну, а також для видалення, з можливістю уловлювання, токсичних речовин, слід здійснити його попередній піроліз. Експериментально вивчено вплив технічного гідролізного лігніну різного ступеня піролізації на процес залізорудної агломерації і властивості отриманого агломерату. Вихідний лігнін піддавався попередній термічній обробці до кінцевої температури 400, 600, 800 і 1000 °C без доступу повітря. Спікання агломерату за участю піролізованого лігніну проводили на лабораторній агломераційній установці. Після спікання визначали міцність агломерату, досліджували його макроструктуру. Хімічний склад зразків агломерату досліджували методом рентгенофлуоресцентного аналізу.

В результаті проведених експериментів визначена можливість заміни 25 % коксового дріб'язку лігніном, попередньо піролізованим при температурі 800 °C. За таких умов основні показники агломераційного процесу, такі як вертикальна швидкість спікання, вихід придатного продукту і питома продуктивність установки, практично не змінюються.

Спостерігається незначне зниження міцності агломерату на удар і на стирання, однак дані показники залишаються на технологічно прийнятному рівні. Слід зазначити, що при використанні лігніну в якості агломераційного палива виявляється тенденція до деякого зниження вмісту заліза в агломераті. Дослідження макроструктури агломерату показало збільшення діаметра пор при частковій заміні коксового дріб'язку лігніном, причому з підвищенням температури піролізу лігніну, обсяг пор збільшується.

Проведені дослідження підтвердили можливість вирішення актуальної екологічної проблеми утилізації технічного лігніну, шляхом застосування його в агломераційному процесі з попередньою його піролізацією. Перспективним напрямком подальших досліджень є розвиток способів підготовки технічного гідролізного лігніну до використання в залізорудній агломерації

Ключові слова: утилізація промислових відходів, технічний гідролізний лігнін, піроліз, залізорудна агломерація

1. Introduction

Technical hydrolysis lignin (THL) is an industrial waste that is formed at the enterprises of the hydrolysis, in pulp and paper industry as a result of chemical treatment of wood biomass and substandard agricultural raw materials. Currently, the estimated accumulation of THL is about 70–90 million tons in Russia [1], 5–15 million tons in Ukraine [2], 4.3 million tons in Belarus [3]. The land resources alienated for hydrolysis dumps do not subject to reclamation, and THL serves as a source of various hydrocarbon compounds in the environment, including toxic furfural [4]. The analysis performed [5] shows the presence of phenols, phenanthrene and methanol in the lignin samples. The highest total content of organic compounds is observed in the lignin samples taken directly on production. Phenols prevail in the samples of the stored lignin of the upper horizons, methanol – in the lower ones. The results indicate the penetration of formaldehyde and phenols in aquifers.

Industrial methods of utilization of waste lignin in the required amount are not used. A certain part of THL is used as household fuel, filler, burning additives and a component for the production of carbon materials and sorbents. However, the share of lignin processed in this way is small. According to the EUROLIGNIN international organization, just over 1 million tons of practically used lignin account for 50 million tons of THL annually extracted from plant raw materials in the world [6]. Therefore, processing of THL is an urgent issue.

2. Literature review and problem statement

The perspectives of technical hydrolysis lignin utilization in relation to various technological processes have been studied.

The results of the study [7] on the use of lignin in the form of lignin sulfate in the particle board industry are presented. It is shown that the parameters of the obtained materials are comparable with the parameters of traditional materials made from phenol-formaldehyde resins, but there remain unresolved issues related to the purification of technical hydrolysis lignin for its use in the proposed technology. The difficulties of preliminary purification of technical lignin, as well as the presence of harmful substances, do not allow using its considerably in the proposed technology. An option to overcome these difficulties can be in application for other directions. The possible areas can be production of nanomaterials [8], dyes [9], and wastewater treatment [10]. Preparing nanospheres from lignin previously obtained in the paper industry is considered in [8]. The proposed technology is technically simple and cost-effective, however, it can be applied only for newly formed lignin, since lignin accumulated in dumps has a significant amount of impurities. In addition, the production volume of nanospheres will not allow the utilization of all the formed lignin. The study [9] concentrates on using lignin possibility in textile, industry paint and varnish industries, as well as wastewater treatment, is investigated. In another work [10] the possibility of lignin application as a sorbent for wastewater treatment from zinc have been considered. Nevertheless, there are unresolved issues related to the cleaning of a sorbent after use. The content of harmful substances in it is higher than in the initial lignin and subsequent disposal requires additional costs.

Considering the above, it becomes obvious that a consumer needs for the complete utilization of hydrolysis waste and their demand will be commensurating with the volumes of THL generation. Metallurgy, where lignin can be used as a substitute for solid fuel, can become such a consumer. The effect of replacing part of solid fuel during sintering with various biomaterials is investigated. Accordingly, the study [11] of the effect of replacing coke breeze with charcoal, pine and oak sawdust is carried out. It is shown that when using sawdust materials as a solid fuel, considerable fluctuations in the process parameters are observed. This can be caused by significant volatile content and requires additional research. Gan et al. [12] reported about how the effect of replacing solid fuel with charcoal, pyrolyzed straw and sawdust pellets act in the sintering process. It is found that 40 % of coke breeze can be replaced with charcoal, 20 % and 15 % with pyrolyzed straw and sawdust pellets, respectively. There is a significant increase in the allowable amount of the biomaterials to be added when an increase in the pyrolization degree, but the issues concerning the problem how the sinter yield reduces due to the structural changes of the cake with biomaterials remains unresolved. For this, it is necessary to investigate the structure of the sinter obtained with the addition of biomaterials. However, Mezibricky et al. [13] reported that this approach has showed no difference within phases and compositions of these phases, in the study of the microstructure in the vicinity of pores left by biofuel particles. A possible reason for the structural change, which occur in the sinter with adding biomaterials, is thermal conditions that differ from these when using coke breeze and biomaterials.

The addition of biomaterials, including lignin, into the sintering mixture substantially changes the mechanism and kinetics of sintering processes. At the same time, there are problems associated with determining the optimal amount and the properties of lignin. The influences of technical hydrolysis lignin addition on the processes of fuel combustion and materials sintering are not sufficiently studied. All this suggests that a study on the effect of pyrolyzed THL on the sintering process and sinter properties is advisable.

3. The aim and objectives of the study

The aim of the work is to assess the possibility of partial replacement of coke breeze in the sintering mixture with prepyrolyzed technical lignin.

To achieve the aim, the following objectives have been set:

– to experimentally study the effect of adding THL with various pyrolization degrees on the process of iron ore sintering;

– to study the effect of adding THL of various pyrolization degrees on the composition and properties of the obtained sinter;

– to determine temperature parameters of the pyrolization process of technical hydrolysis lignin for its subsequent use in sintering.

4. Materials and methods for studying the effect of lignin addition on the sintering process

4. 1. Preparation of materials and sintering

To obtain the sinter, the materials provided by the Dnieper Metallurgical Combine, PJSC (Ukraine) were used. The composition of the iron-containing and fluxing components of the sintering mixture is given in Table 1.

Table 1

Composition of the iron-containing and flux components of the mixture

Materials	Content, %							
	Fe_{total}	FeO	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO	LOI
Iron ore concentrate	65.88	28.27	62.71	6.44	0.30	0.17	0.26	1.85
Iron ore	57.75	1.76	80.54	12.71	1.60	1.72	0.62	1.05
Limestone	0.28	–	0.40	1.5	0.56	51.5	0.92	45.12
Lime	0.12	–	0.18	1.8	0.26	86.8	1.40	9.44

The initial lignin was used in the form of pillow-shaped briquettes prepared via roll press [12]. The briquettes were subjected to preliminary heat treatment to different final temperatures (400, 600, 800 and 1000 °C) without air access in the electric pit-type furnace. The thermal holding time at the final temperature was 20 minutes. After pyrolysis, the retort with the residue was taken out of the furnace and dry-quenched. The resulting material was crushed to the size of less than 7 mm. Exceeding the specified size limit is impractical as it can lead to the appearance of a local re-melted sinter cake.

The properties of coke breeze and lignin were determined additionally. In the experiments, technical hydrolysis lining of the Zaporizhzhya hydrolysis-yeast plant was used. The technical properties of coke breeze are presented in Table 2, proximate and ultimate analyzes of the initial and the pyrolyzed lignin in Table 3.

Table 2

Technical properties of coke breeze

Moisture, %		Ash content on dry basis, %	Volatile matter, %		Total sulfur on dry basis, %	kJ/kg
as received W^r	air dried basis W^a	dry basis A^d	on dry basis V^d	dry-ash free basis V^{daf}	S_t^d	Law Calorific Value
1.3	0.9	11.7	3.5	3.9	0.32	27013

Table 3

Proximate and ultimate analyzes of the initial and the pyrolyzed lignin

Fuel (pyrolysis temperature, °C)	Moisture W^a, %	Ash content A^d, %	Volatile matter V^d/V^{daf}, %	Total sulfur S_t^d, %	Hydrogen H^d, %	Carbon C^d, %	Nitrogen N^d, %
Lignin (in.)	7.8	33.4	41.8/62.9	1.04	3.56	52.40	0.69
Lignin (400)	4.1	40.1	18.7/31.2	1.00	2.10	47.87	0.67
Lignin (600)	2.6	45.8	8.4/15.5	0.99	1.29	49.35	0.66
Lignin (800)	2.1	50.9	3.4/6.9	1.03	0.71	46.23	0.55
Lignin (1000)	6.7	48.9	5.7/11.1	1.07	0.61	48.66	0.45

Sintering was performed via lab-scale sinter pot: the sintering parameters and the mixture composition corresponded to the real sintering mixture of the Dnieper Metallurgical Combine, PJSC. Additionally, an increase in heat losses in the lab-scale sinter pot as compared with the industrial units was taken into account. The composition of the sintering mixture is shown in Table 4. The amount of THL pyrolyzed to different temperatures in all experiments was 25 % of the total amount of solid fuel used. Generally, it is not recommended to exceed this value since it can lead to an excessive reduction in the total calorific value of the solid fuel mixture.

Table 4

Composition of the sintering mixture

Components	Content, %	
	Without lignin	With lignin
Concentrate	46.75	46.75
Iron ore	10.5	10.5
Lime	1.5	1.5
Limestone	10.25	10.25
Coke breeze	6	4.5
Pyrolyzed lignin	–	1.5
Return	25	25
Total	100	100
Moisture	8	8

4. 2. Sinter strength assessing method and equipment for studying its macrostructure and chemical composition

After sintering, the impact strength of the sinter (fraction yield +5 mm after tests) and abrasion strength (fraction yield less than 0.5 mm after tests) has been determined.

The study of the sinter macrostructure has been performed using the Neophot-21 inverted reflected light photomicroscope.

The chemical composition of the studied samples has been studied by X-Ray Fluorescence analysis using the EXPERT 3L precision analyzer. The detection limit of elements for 100 s (from 12 Mg to 92 U) is ≤0.05 %. The detector applied was SDD with a nominal statistical loading of the spectroscopic path of 52,000 s^{-1} and the resolution (for KαMn) at nominal loading less than 149 eV.

5. Study results: the effect of adding prepyrolyzed technical hydrolysis lignin on the iron ore sintering process

Table 5 shows the principle results of the sintering. In the case when lignin pyrolyzed at temperatures of 400 and 600 °C was applied, the required amount of the sinter to determine strength characteristics was not obtained.

Fig. 1, 2 show the effect of the pyrolization degree of lignin on the sinter yield and vertical sintering rate in comparison with the application of only coke breeze as a fuel.

Table 5

Results of sintering with lignin of different pyrolization degrees

Process parameters	Fuel				
	Coke breeze	Lignin			
Pyrolysis temperature, °C	1050	400	600	800	1000
Sinter yield, %	69.25	41.75	37.5	66.75	51.75
Impact strength, %	94	–	–	90.5	77
Abrasion strength, %	0.3	–	–	0.5	1
Vertical sintering rate, mm/min	32.78	22.31	25.2	29.5	20.29
Specific capacity, t/m²·h	1.84	0.77	0.78	1.59	0.88

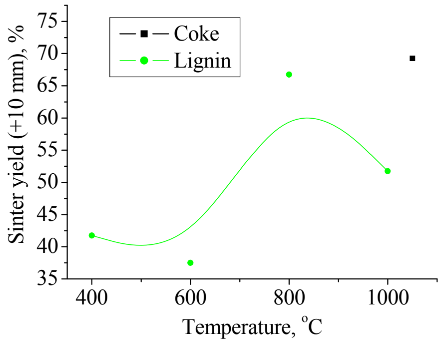

Fig. 1. Dependence of sinter yield (+10 mm) on the lignin pyrolysis temperature

It is found that when using lignin at the pyrolization temperatures of 400 and 600 °C, the sinter yield and quality are significantly reduced. In turn, the usage of lignin prepyrolyzed at 800 °C allows obtaining indicators of the sintering process at a basic level. At the same time, the impact and abrasion strengths of the sinter have been reduced slightly compared with the standard one (only coke breeze). Increasing the pyrolization temperature up to 1000 °C lead to neither an improvement of sintering parameters nor sinter properties.

The microstructures of the sinters obtained only with coke breeze and those with the mixtures of coke breeze plus THL prepyrolyzed to 800 and 1000 °C are shown in Fig. 3. The obtained sinters have a complex structure and in the case of using THL are characterized by increased diameter of pores and a number of cracks.

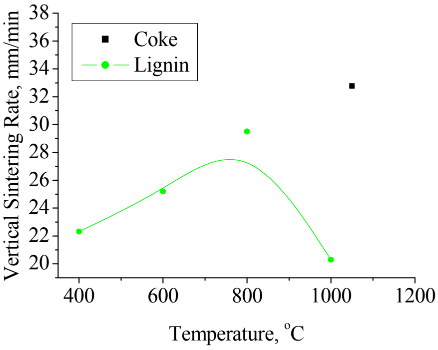

Fig. 2. Dependence of the vertical sintering rate on the THL pyrolysis temperature

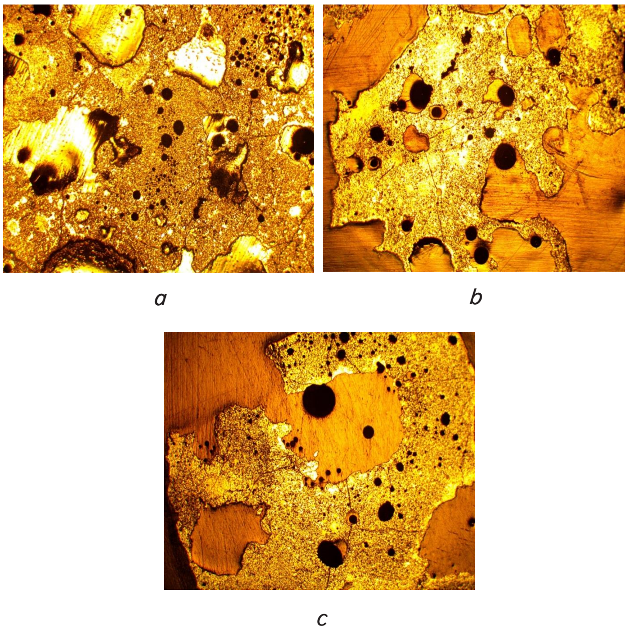

Fig. 3. Macrostructures of the obtained sinters (×10): *a* — with coke breeze; *b* — with lignin pyrolyzed at 800 °C; *c* — with lignin pyrolyzed at 1000 °C

Table 6 shows the elemental composition of the sinters according to X-Ray Fluorescence analysis. Fig. 4 illustrates the XRF spectra of the sinters obtained using coke breeze and its mixtures with lignin of different pyrolization degrees as solid fuel.

The application of THL as a fuel causes the decrease in the iron content within the sinter due to the higher ash content of THL compared to coke. The dramatical increase in the silicon content and the decrease in the aluminum content in the sinters obtained with THL should also be noted.

Table 6

Elemental composition of the sinters

Fuel (pyrolysis temperature, °C)	Basicity, CaO/SiO_2	Content of elements, %					
		Al_2O_3	SiO_2	CaO	Mn	Fe_{total}	O_{total}
Coke	1.19	0.992	10.209	12.103	–	53.641	32.402
Lignin (800)	0.86	0.703	14.848	12.715	0.12	50.001	33.449
Lignin (1000)	0.83	0.927	13.847	11.453	0.039	51.102	33.417

6. Discussion of the study results of the pyrolyzed lignin effect on the sintering process

The proximate analysis of lignin pyrolyzed at different temperatures allows concluding that at the pyrolization temperature of up to 600 °C, lignin contains a large amount of volatiles. This leads to the fact that during the mixture heating such material first gives off volatile substances that do not burn and, consequently, they are carried away by flue gases. At the pyrolysis temperature of 600 °C, an increase in the vertical sintering rate up to 25.2 mm/min is observed. With regard to the insufficient amount of the heat entering the layer, the sintering process is not fully completed and the sinter yield decreases to 37.5 %. Taking into account that THL after pyrolysis at 800 °C has relatively low volatile substances, this fuel provides the best parameters of the sintering process, comparable to the parameters when using coke breeze and allows achieving specific capacity of the sinter pot of 1.59 t/m^2·h. In the case of using THL of the pyrolization temperature of 1000 °C, faster fuel burnout, layer remelting and, as a result, decrease in the vertical sintering rate to 20.29 mm/min are observed. This is due to the greater reactivity of THL after pyrolysis, compared with coke, which is consistent with above-mentioned studies [7, 8]. The sintering process in such conditions is better than using lignin with the pyrolization temperature of 600 °C and the yield makes 51.75 %. However, in both cases, there is a low specific capacity of the sinter pot – 0.78 t/m^2·h at the pyrolysis temperature of 600 °C and 0.88 t/m^2·h at 1000 °C.

That, as the THL pyrolization temperature increases from 800 to 1000 °C, an increase in the maximum diameter and the total number of the sinter pores is observed. This can be explained by the fact that due to the higher reactivity of the pyrolyzed lignin, there should be a higher temperature at the place of its location within the layer. This, in turn, is to lead to the sinter re-melting.

Additionally, the particle size of lignin is up to 7 mm, compared with the coke breeze size (up to 3 mm). Increasing the particle size is necessary for achieving the same time of fuel combustion in the layer horizon, based on increased reactivity of pyrolyzed lignin.

Moreover, the use of THL with the pyrolization temperature of 800 °C allows increasing the sinter porosity with a slight decrease in strength. On the other hand, with an increase in the lignin pyrolysis temperature, the average maximum pore diameter increases from 4.14 mm at the pyrolization temperature of 80 °C to 4.87 mm at 1000 °C. In addition, an increase in the reducing ability of the sinter during blast furnace smelting is expected. Under conditions of lignin industrial use, the reduction of the iron content in the sinter will not exceed 0.5 % if replacing 25 % of solid fuel. There will be lower fuel consumption compared with the lab-scale sinter pot.

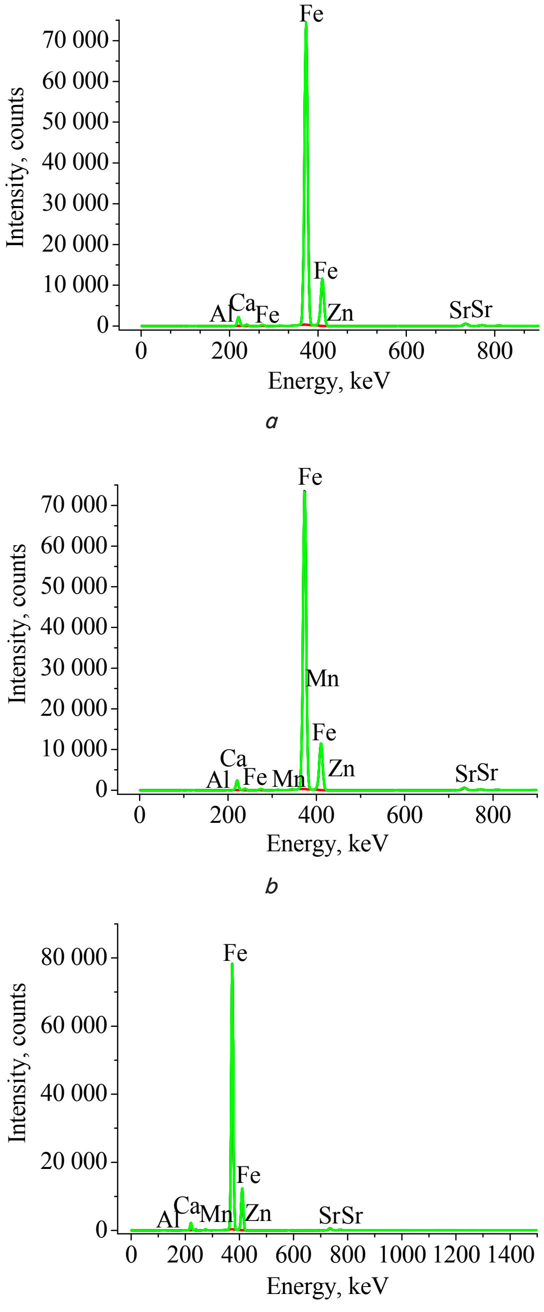

Fig. 4. XRF spectra of the obtained sinters: *a* – with coke breeze; *b* – with lignin pyrolyzed at 800 °C; *c* – with lignin pyrolyzed at 1000 °C

The obtained results are consistent with [12], where the permissible amount of biomaterials added to the sintering mixture is established according to the degree of their pyrolization. However, unlike the results published in [12], the analysis of the effect of lignin pyrolization degree on the sintering process suggests the following:

– the volatiles contained in the fuel do not completely burn during sintering, their heat is not used in the sintering process, therefore the minimum volatile content is necessary;

– for uniform heat treatment of the sintered layer using pyrolyzed lignin, it is necessary to achieve the lignin burning rate equal to the burning rate of coke breeze.

These conditions are achieved by selecting the pyrolization temperature, as well as the size and density of the particles of the initial lignin.

The relevant direction of further work is the development of methods for preparing technical hydrolysis lignin for utilization in iron ore sintering as solid fuel. Since THL is a fine-grained bulk material, its application requires preliminary pelletizing, that can be carried out by roll press briquetting [14] or by forced spinneret granulation [15].

7. Conclusions

1. The effect of technical hydrolysis lignin, subjected to pyrolization at temperatures of 400, 600, 800 and 1000 °C, on the process of iron ore materials sintering and the properties of the obtained sinter has been experimentally studied in the publication. The replacement of coke breeze up to 25 % by lignin pyrolyzed at 800 °C in iron-ore sinter charge sustained in the efficiency of the sintering process, which remained at an acceptable level of 1.59 $t/m^2 \cdot h$. On the other hand, the impact strength and abrasion strength values of the sinter decrease slightly by 3.5 % and 0.2 %, respectively.

2. The study of the sinter macrostructure has showed an increase in the pore diameter when using lignin. At the same time, with an increase in the lignin pyrolysis temperature, the average maximum pore diameter increases from 4.14 mm at the pyrolization temperature of 800 °C to 4.87 mm at 1000 °C.

3. Thus, a number of studies reveals the prospects of the proposed direction of processing technical hydrolysis lignin by using it as a solid fuel in iron ore sintering. It is found that the maximum lignin pyrolization temperature should be 800 °C.

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