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Д.В. Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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NEWS

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NAS RK is pleased to announce that News of NAS RK. Series of chemistry and technologies scientific journal has been accepted for indexing in the Emerging Sources Citation Index, a new edition of Web of Science. Content in this index is under consideration by Clarivate Analytics to be accepted in the Science Citation Index Expanded, the Social Sciences Citation Index, and the Arts & Humanities Citation Index. The quality and depth of content Web of Science offers to researchers, authors, publishers, and institutions sets it apart from other research databases. The inclusion of News of NAS RK. Series of chemistry and technologies in the Emerging Sources Citation Index demonstrates our dedication to providing the most relevant and influential content of chemical sciences to our community.

Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Web of Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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OBTAINING BIOCHAR FROM RICE HUSK AND STRAW

Abstract. This paper presents the results of research on obtaining biochar from agricultural plant waste such as rice husk and straw. The selection of the optimal conditions for thermolysis, such as the duration and temperature of the process, has been conducted. The thermolysis products are characterized for iodine adsorption activity, cumulative water pore volume, and for bulk density. The porous structure of the obtained products has been studied by scanning electron microscopy. Based on the results of the research conducted, it has been found that biochars obtained from husk and straw with a thermolysis duration of 30 minutes have low iodine sorption characteristics and water pore volumes. With an increase in the duration of thermolysis, the sorption characteristics improve, the optimal for the husk is the thermolysis duration of 60 minutes at a temperature of 500°C, and for straw, the optimal thermolysis duration is 60 minutes at a temperature of 300°C. The best option is biochar obtained from rice straw at a duration of 60 minutes and a thermolysis temperature of 300°C, having an iodine adsorption activity of 54.61%, a cumulative water pore volume of 0.941 cm³/g and a bulk density of 169.29 g/dm³. The obtained biochars from rice husk and straw have been studied by scanning electron microscopy at 4300 and 5000 times magnification, and they have a developed porous structure.

According to the literature, it is known that biochar can also be used as a renewable energy source. Research has been carried out to determine the calorific value of the obtained biochars. To compare the calorific value of rice husk, straw and the obtained biochars, their heating values have been determined on a calorimeter. The highest heating value has a biochar obtained from husk at a duration of 60 minutes and at a thermolysis temperature of 400°C with a value of 17.520 kJ/g, the optimal for biochar obtained from straw is a duration of 60 minutes and a thermolysis temperature of 400°C with a value of 16.451 kJ/g.

The experimental data obtained make it possible to use the obtained biochar from rice straw in the future as a biofertilizer to improve the characteristics of soils, as well as to use biochar obtained from rice husk to produce renewable fuel.

Key words: biochar, rice husk, rice straw, thermolysis, recycling, fertilizer, renewable fuel.

Introduction. Rice husk and straw are large-tonnage agricultural wastes that are disposed of by incineration; this waste requires special attention for processing. The processing of this waste into a useful product – biochar is especially relevant for rice-growing regions.

Biochar is a promising biofertilizer obtained from plant waste by thermolysis. The application of this fertilizer gives many positive qualities to soils, and due to its porous structure, it allows to retain moisture, macro- and microelements in the form necessary for plants, reduces soil salinization, prevents fertilizers from washing out and accumulates them in their structure, helps plant roots to assimilate nutritional substances, forwards the growth of plants, prevents aggregation of soil into lumps, prevents soil crust formation, increases crop yield up to 40% and the soil fertilized with biochar preserves its fertility for several decades, and in some cases for centuries.

The world production of rice is more than 750 million tons per year, during the harvesting and processing of rice, a large amount of waste is generated in the form of rice husk and straw, the proportion of these waste is 20% and 50%, respectively. Currently, the abovementioned waste is not recycled and disposed of by incineration, polluting the environment, and rice husk also contains a large amount of silicon, when it is burned, finely dispersed silicon dioxide is formed, which in turn exposes people to a dangerous incurable disease – silicosis.

Biochar is a new direction in science, the first scholarly works on its obtainment from organic waste were published in the early 2000s, but basically for obtaining renewable fuels and adsorbents [1-5], the first works on the use of biochar in agriculture as fertilizer appeared in 2006-2007 [6-10]. Research in this direction has not been carried out by Kazakh scientists, the use of biochar in Kazakhstan is pioneering. When sowing and growing rice, a high salinization, washout of useful soil elements, etc. is observed. We propose the processing of rice waste into biochar and its application to improve the characteristics of soils. The authors of the paper have a groundwork on the thermal processing of plant waste and the obtainment of biochar and coal sorbents from them, which have high sorption characteristics [11-17].

The paper provides data that the use of biochar together with nitrogen fertilizers in rice sowing increases the yield of rice up to 44.4% [18]. Biochar obtained from rice straw normalizes the pH of acidic soil, reduces N₂O emissions into the environment by 83% and activates mineral nitrogen [19]. The authors have studied the nature of sorption/desorption of biourea by biochar obtained from rice husk and straw at different pyrolysis temperatures (300, 450, 600°C), it has been found that the maximum adsorption of urea has been shown by biochar from rice straw and husk obtained at a pyrolysis temperature of 450°C, and these composites have the potential to enhance agricultural yield through the efficient use of nitrogen. Also, the authors estimates that a single introduction of this biochar into the soil makes it possible to use this soil without adding biochar for 500-750 years [20]. The paper presents a comparative analysis of the effect of biochar obtained from sasanqua camellia seed shells and rice straw on the N₂O emission of nitrogen fertilized rice lands and their water-retaining capacity. Soil treated with biochar obtained from rice straw reduces N₂O emission by 363%, biochar from sasanqua camellia seed shells by 200%, and their water-retaining capacity is 120% and 70%, respectively [21]. Biochar was used to clean oil-contaminated soils in a composition with fertilizers, which in turn made it possible to reduce the bulk density, improve air exchange, provide favorable conditions for microbial activity, thus oil-contaminated soil treated with fertilizer without adding biochar is cleaned in 230 days, and in combination with biochar in 30 days [22-28]. The paper provides data on the effect of biochar for six years (2009-2015) on characterization of soil and rice crop. The soil was treated with biochar at a rate of 10 t/ha, compared to untreated soil, the organic carbon content increased by 45%, the total available nitrogen reserve by 30%, the mass median diameter of water resistant fillers increased by 25%, the nitrogen of microbial biomass and enzyme activity increased by 30%, bulk density decreased, rice crop was 10% higher [29]. When the soil is treated with biochar at doses from 4.5 to 40 t/ha, a significant decrease in the thermal conductivity of the soil is observed, which in turn has a positive effect on the growth and development of plants [30]. There is data on the immobilization of cadmium with biochar in the soil, thus when the soil is treated with unmodified and KOH modified biochar at a dose of 30 g/kg, there is a decrease in the soluble fraction of cadmium by 30.3% and 27.4%, bioavailable cadmium by 32.4% and 25, 2%, respectively [31]. Research has been carried out on the application of biochar into the soil at rates of 6-12 kg/m² in the autumn, spring and mixed seasons and the effect on the growth and productivity of soybeans was studied, so when applying biochar of 9 kg/m² into the soil in the spring-autumn period is favorable for growth of soybeans, the height increases, metabolism accelerates and the diameter of the stem decreases imperceptibly [32]. There is data on the effect of the treated soil with biochar on the growth of rice when irrigated with saline water. Three experiments have been carried out with the application of biochar into the soil in the ratio of 33.75 t/ha, 67.5 t/ha and 101.25 t/ha, based on the research findings it has been found that the addition of biochar reduces significantly sodium ions in water, but at the same time, sodium ions remain unchanged, such indicators as the water status of leaves, chlorophyll content index, rice biomass increase and, accordingly, rice crop have improved. The best indicators have been achieved at a rate of 67.5 t/ha [33]. Research has been carried out on the effect of biochar from rice straw, cow manure and their combination modified with ZnO nanoparticles at rates of 8 t/ha on the yield of sunflowers grown on agricultural lands irrigated for a long time with polluted wastewater. The best indicators are in the combination of biochar rice straw: cow

manure = 50:50, the availability of heavy metals in the soil such as Pb, Cr, Cu and Cd has decreased by 78.6%, 115.3%, 153.3% and 178.5%, respectively, compared to untreated lands. Also, decreases the content of Pb, Cr, Cu and Cd in plants to 1.13, 5.19, 3.88 and 0.26 mg/kg of dry substance, and the number of seeds in the head increases by 50.4% [34]. The effect of biochar obtained from walnut on the microbial community in soils during wet and dry cycles has been also studied. It has been found that biochar increases soil pH through moisture, stabilizes the microbial community of the soil [35].

According to the results of literature data on the processing of agricultural wastes into a useful product and their application to improve the characteristics of soils, opens a prospect for further research in this topical area.

Materials and methods. Thermolysis of rice husk and straw was carried out in a BR-12NFT tube furnace (China) at a temperature of 300-500°C in the nitrogen atmosphere. The iodine adsorption activity, cumulative water pore volume, and bulk density of the obtained products were determined by standard methods.

To determine the iodine adsorption activity, a solution of iodine in potassium iodide at a concentration of 0.1 mol/dm³ was added to the suspended part of the biochar and agitated in an EKROS 6300 mixer (Russia) for 15 minutes at an intensity of 100-125 vibrations per minute. Then the solution was allowed to settle, and the required volume of solution was taken from the flask with a pipette for further titration with a solution of 0.1 mol/dm³ sodium thiosulfate, using a starch solution as an indicator, until the blue color disappeared [36].

The cumulative water pore volume was determined by filling the pores with water from 0.5 to 104 nm by boiling coal charge in water for 15 minutes and disposal of excess water from the grain surface by suction at a vacuum of 8 kPa and further weighing [37].

The bulk density of biochar was determined by measuring the mass of biochar occupying a certain volume at normalized compaction [38].

The microphotography of the obtained biochars was taken on a JSM-6510LV Scanning Electron Microscope by Jeol (Japan).

The calorific value of biochar was determined on a C2000 calorimeter by Ika-Werke (Germany).

Results and discussion. 30 g of rice husk or straw were placed in a tube furnace, and sealed, the tube was filled with gaseous nitrogen supplied from a balloon, and the thermolysis process was carried out with a rate of temperature elevation of 10°C per minute up to 300-500°C and kept at these temperatures for 30-60 minutes. The influence of temperature and duration of the thermolysis process on the yield and characteristics of biochar were determined (table 1).

Table 1 – Influence of duration and temperature of thermolysis on the yield and characteristics of biochar

Raw material	Duration, min	Temperature, °C	Yield of the obtained product, % wt	Iodine adsorption activity, %	Cumulative water pore volume, cm ³ /g	Bulk density, g/dm ³
Husk	30	300	55,54	17,78	0,373	338,52
		400	54,52	15,24	0,367	338,14
		500	35,59	10,16	0,336	295,59
	60	300	51,75	12,70	0,386	304,51
		400	47,10	15,17	0,392	287,21
		500	40,52	19,05	0,402	266,19
Straw	30	300	48,82	21,59	0,978	114,70
		400	40,74	13,97	0,957	109,37
		500	34,52	11,43	0,879	98,97
	60	300	51,04	54,61	0,941	169,29
		400	47,82	38,10	0,762	129,67
		500	41,82	22,86	0,746	110,12

It can be seen from the table that biochars obtained from husk and straw with a thermolysis duration of 30 minutes have low iodine sorption characteristics and water pore volumes. With an increase in the duration of thermolysis, the sorption characteristics improve, thus the optimal for the husk is the thermolysis duration of 60 minutes at a temperature of 500°C, and for straw, the optimal thermolysis duration is 60 minutes at a temperature of 300°C.

The best option is biochar obtained from rice straw at $\tau = 60$ minutes and $t = 300^\circ\text{C}$, having an iodine adsorption activity of 54.61%, a cumulative water pore volume of $0.941 \text{ cm}^3/\text{g}$ and a bulk density of $169.29 \text{ g}/\text{dm}^3$. Figure 1 shows a photograph of biochar obtained from rice straw.



Figure 1 – Photograph of biochar obtained from rice straw

The obtained biochars from rice husk and straw have been studied by scanning electron microscopy at 4300 and 5000 times magnification. The microphotographs show the developed porous structure of biochar obtained from rice husk and straw (figures 2 and 3).

The experimental data obtained make it possible to use the obtained biochar from rice straw in the future as a biofertilizer to improve the characteristics of soils.

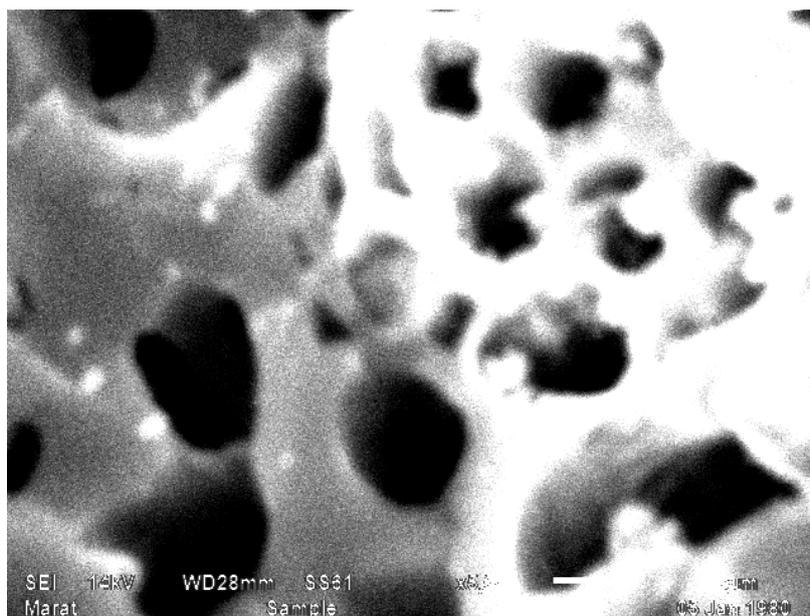


Figure 2 – Microphotograph of biochar obtained from rice husk

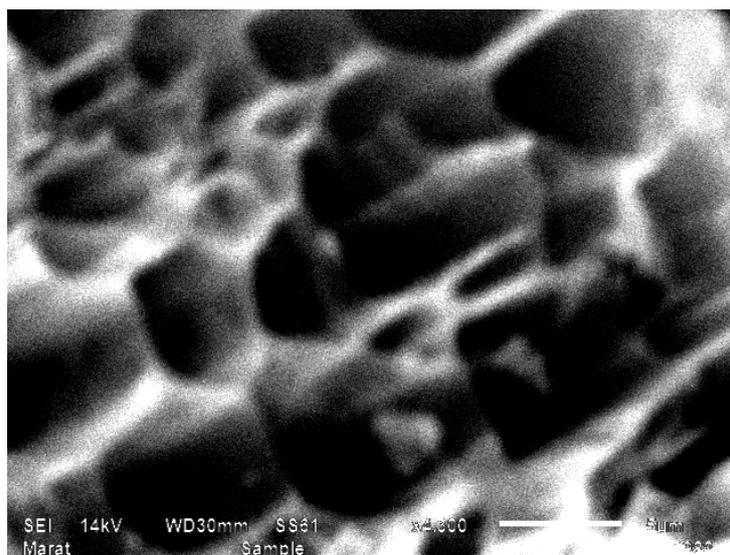


Figure 3 – Microphotograph of biochar obtained from rice straw

As mentioned above, biochar can also be used as a renewable energy source, we have carried out studies to determine the calorific value of the obtained biochars. To compare the calorific value of rice husk, straw and the obtained biochars, their heating values have been determined on a calorimeter; the data are shown in table 2.

Table 2 – Study of the calorific value of rice husk, straw and biochar obtained from them

Description	Thermolysis duration, min	Thermolysis temperature, °C	Heating values, kJ/g
Rice husk	–	–	11,152
Rice straw	–	–	12,669
Rice husk biochar	60	300	15,720
		400	17,520
		500	15,908
	30	300	10,792
		400	17,184
		500	16,257
Rice straw biochar	60	300	14,036
		400	16,451
		500	11,900
	30	300	12,589
		400	14,183
		500	13,905

The highest heating value has a biochar obtained from husk at $\tau = 60$ minutes and $t = 400^\circ\text{C}$ with a value of 17.520 kJ/g, the optimal for biochar obtained from straw is $\tau = 60$ minutes and $t = 400^\circ\text{C}$ with a value of 16.451 kJ/g. The data also make it possible to use biochar to obtain fuel pellets that have higher calorific value than rice husk and straw.

Conclusions. Thus, biochars were obtained by thermolysis of rice husk and straw at a duration of 30-60 minutes and at temperatures of 300-500°C. The optimal is biochar obtained from rice straw at $\tau = 60$ minutes and $t = 300^\circ\text{C}$, having an iodine adsorption activity of 54.61%, a cumulative water pore

volume of 0.941 cm³/g and a bulk density of 169.29 g/dm³. Biochar obtained from rice husk at $\tau = 60$ minutes and $t = 500^\circ\text{C}$ has higher characteristics compared to other options obtained from husk with an iodine adsorption activity of 19.05%, a cumulative water pore volume of 0.402 cm³/g and a bulk density of 266.19 g/dm³. The calorific value of the obtained products has been studied, the most optimal is biochar obtained from husk at $\tau = 60$ minutes and $t = 400^\circ\text{C}$ with a value of 17.520 kJ/g. The research results make it possible to use the biochar obtained under optimal conditions as a biofertilizer to improve the characteristics of soils, as well as to produce renewable fuel.

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КҮРІШ ҚАУЫЗЫ МЕН САБАНЫНАН БИОЧАР АЛУ

Аннотация. Күріш қауызы мен сабаны көптонналы ауылшаруашылық қалдығы болып саналады, қазіргі уақытта өртеу арқылы утилизациялануына байланысты оларды өңдеу жұмыстары өте маңызды. Термолиздің оңтайлы жағдайларын таңдау, мысалы, процестің ұзақтығы мен температурасы. Термолиз өнімдері йодтың адсорбциялық белсенділігімен, су саңылауларының жиынтық көлемімен және сусымалы тығыздығымен сипатталады. Сканерлейтін электронды микроскопия әдісімен алынған өнімдердің кеуекті құрылымы зерттелді.

Жүргізілген зерттеу нәтижелері бойынша қауыз бен сабаннан 30 минут термолиз ұзақтығында алынған биочар йод бойынша төмен сорбциялық қасиеттерге ие және су бойынша кеуек көлемінің төмен екендігі анықталды. Термолиз ұзақтығын арттырғанда сорбциялық сипаттамалар жақсарады, күріш сабанын 60 мин ұзақтықта және 300°C температурада термолиздегенде, алынған биочар оңтайлы болып саналады, бұл жағдайда алынған өнімнің йод бойынша адсорбциялық белсенділігі 54,61%, су бойынша кеуектің жинақ көлемі 0,941 см³/г және үйінді тығыздығы 169,29 г/дм³ құрайды. Күріш қауызы мен сабанынан алынған биочарлар растрлы электронды микроскопия әдісімен 4300 және 5000 есе ұлғайтылып зерттелді, олардың жоғары кеуекті құрылымға ие екендігі анықталды.

Әдеби мәліметтерде биочарды жаңартылатын энергия көзі ретінде қолдануға болатындығы белгілі. Алынған биочарлардың жылу бөлу мүмкіндіктерін анықтау бойынша зерттеулер жүргізілді. Күріш қауызы, сабаны және алынған биочарлардың жылу бөлу мүмкіндігін салыстыру үшін олардың жану жылуы С2000 калориметрде анықталды. 60 мин ұзақтықта және 400°C температурада күріш қауызын термолиздеу арқылы алынған биочар жану жылуының ең жоғары энергиясына ие, жану жылуы – 17,520 кДж/г тең, күріш сабаны үшін жоғарғы көрсеткіш те аталған ұзақтық пен температура болып саналады, жану жылуы – 16,451 кДж/г тең.

Алынған тәжірибелік мәліметтер күріш сабанынан алынған биочарды алдағы уақытта топырақ сипаттамаларын арттыратын биотыңайтқыш ретінде, ал қауыздан алынған биочарды жаңартылатын отын ретінде қолдануға мүмкіндік береді.

Түйін сөздер: биочар, күріш қауызы, күріш сабаны, термолиз, қайта өңдеу, тыңайтқыш, жаңартылатын отын.

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ПОЛУЧЕНИЕ БИОЧАРА ИЗ РИСОВОЙ ШЕЛУХИ И СОЛОМЫ

Аннотация. В работе представлены результаты исследований по получению биочара из отходов сельскохозяйственных растений, таких как рисовая шелуха и солома. Проведен выбор оптимальных условий термолиза, таких как продолжительность и температура процесса. Продукты термолиза характеризуются адсорбционной активностью йода, кумулятивным объемом пор воды и насыпной плотностью. Методом

сканирующей электронной микроскопии исследована пористая структура полученных изделий. По результатам проведенных исследований установлено, что биочары, полученные из шелухи и соломы с длительностью термоллиза 30 мин, обладают низкими сорбционными характеристиками йода и объемом пор воды. С увеличением продолжительности термоллиза улучшаются сорбционные характеристики, оптимальной для шелухи является продолжительность термоллиза 60 мин при температуре 500°C, а для соломы оптимальной продолжительностью термоллиза является 60 мин при температуре 300°C. Оптимальным вариантом является биочар, полученный из рисовой соломы длительностью 60 мин и температурой термоллиза 300°C, обладающий адсорбционной активностью йода 54,61%, совокупным объемом пор воды 0,941 см³/г и насыпной плотностью 169,29 г/дм³. Полученные биочары из рисовой шелухи и соломы исследованы методом сканирующей электронной микроскопии при увеличении в 4300 и 5000 раз и имеют развитую пористую структуру.

Согласно литературе, известно, что биочар также может быть использован в качестве возобновляемого источника энергии. Проведены исследования по определению теплотворной способности полученных биочаров. Для сравнения теплотворной способности рисовой шелухи, соломы и полученных биочаров были определены их теплотворные способности на калориметре. Наибольшей теплотворной способностью обладает биочар, полученный из шелухи при продолжительности 60 мин и температуре термоллиза 400°C со значением 17,520 кДж/г, оптимальной для биочара, полученного из соломы, является продолжительность 60 мин и температура термоллиза 400°C со значением 16,451 кДж/г.

Полученные экспериментальные данные позволяют использовать полученный биочар из рисовой соломы в дальнейшем в качестве биоудобрения для улучшения характеристик почв, а также использовать биочар, полученный из рисовой шелухи, для получения возобновляемого топлива.

Ключевые слова: биочар, рисовая шелуха, рисовая солома, термоллиз, переработка, удобрение, возобновляемое топливо.

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