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A STUDY OF LIGNIN-FREE RICE HUSK DECOMPOSITION KINETICS

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Abstract

The study addresses the problem of using recycled materials for the production of a wide range of diverse products; in this context, the paper investigates the extraction of amorphous silicon (IV) dioxide from rice waste, i.e. rice husk, which differs in its chemical composition from all other cereal crops by a high content of silicon dioxide. Amorphous silicon (IV) oxide is widely used in electronics, medicine, food industry, cosmetology, paintwork materials manufacturing, and other industries. Amorphous silicon(IV) oxide has to meet various requirements, the main ones being amorphous structure, degree of purification, and particle size. A derivatographic method of analysis is used to study the non-isothermal kinetics of rice husk residue thermal decomposition. According to the results of derivatographic, chemical, and phase analyzes, a method for amorphous silicon (IV) oxide extraction by thermal decomposition of rice husk after the lignin removal has been proposed. The values of relative activation energies and the pre-exponential factors of the reactions have been calculated. A mathematical model characterized by a system consisting of three first order differential equations and four algebraic equations has been designed. Through the use of the proposed model, the time response characteristics of the process have been studied.

Keywords: kinetics, rice husk; mechanism; lignin; decomposition; silicon dioxide

ДОСЛІДЖЕННЯ КІНЕТИКИ ТЕРМІЧНОГО РОЗКЛАДАННЯ РИСОВОГО ЛУШПИННЯ ПІСЛЯ ВИДАЛЕННЯ ЛІГНІНУ

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Анотація

У рамках вирішення проблеми використання вторинної сировини для виробництва широкого спектру різноманітних продуктів, розглядається виділення аморфного силіцій (IV) оксиду з відходів рисового виробництва – рисового лушпиння, яке відрізняється за своїм хімічним складом від усіх інших злакових культур великим вмістом діоксиду кремнію. Аморфний силіцій (IV) оксид має широкий спектр застосування в електроніці, медицині, харчовій промисловості, косметології, при виробництві лаків і фарб, а також в інших галузях промисловості. До аморфного діоксиду кремнію пред'являються різні вимоги, але основними є аморфна структура, ступінь очищення та розмір частинок. Для вивчення неізотермічної кінетики термічного розкладання залишку рисового лушпиння застосовували дериватографічний метод аналізу. За результатами дериватографічного, хімічного і фазового аналізів запропоновано механізм процесу виділення аморфного силіцій (IV) оксиду шляхом термічного розкладання рисового лушпиння після видалення лігніну. Розраховані значення умовних енергій активації і предекспоненціальних множників реакцій. Побудовано математичну модель, яка описується системою, що складається з трьох диференціальних рівнянь першого порядку і чотирьох алгебраїчних. З її допомогою вивчені тимчасові характеристики процесу.

Ключові слова: кінетика; рисове лушпиння; механізм; лігнін; розкладання; силіцій (IV) оксид

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ИССЛЕДОВАНИЕ КИНЕТИКИ ТЕРМИЧЕСКОГО РАЗЛОЖЕНИЯ РИСОВОЙ ШЕЛУХИ ПОСЛЕ УДАЛЕНИЯ ЛИГНИНА

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Аннотация

В рамках решения проблемы использования вторичного сырья для производства широкого спектра разнообразных продуктов, рассматривается выделение аморфного диоксида кремния из отходов рисового производства – рисовой шелухи, которая отличается по своему химическому составу от всех других злаковых культур большим содержанием диоксида кремния. Аморфный диоксид кремния имеет широкий спектр применения в электронике, медицине, пищевой промышленности, косметологии, при производстве лаков и красок, а также в других отраслях промышленности. К аморфному диоксиду кремния предъявляются различные требования, но основными являются аморфная структура, степень очистки и размер частиц. Для изучения неизотермической кинетики термического разложения остатка рисовой шелухи применяли дериватографический метод анализа. По результатам дериватографического, химического и фазового анализов предложен механизм процесса выделения аморфного диоксида кремния путем термического разложения рисовой шелухи после удаления лигнина. Рассчитаны значения условных энергий активации и предэкспоненциальных множителей реакций. Построена математическая модель, которая описывается системой, состоящей из трех дифференциальных уравнений первого порядка и четырех алгебраических. С ее помощью изучены временные характеристики процесса.

Ключевые слова: кинетика, рисовая шелуха, механизм, лигнин, разложение, диоксид кремния

Introduction

One of the important and urgent problems in Ukraine is the disposal of solid wastes and development of environmentally friendly and low-waste technologies of their recycling [1–3]. Large-tonnage, constantly renewable agricultural wastes, namely rice husk (RH) are of great interest. Rice is the most common crop in the world. Rice is grown in more than 100 countries around the world. World rice production is 530 million tons, which corresponds to 110 million tons of rice husk. The countries with the highest level of rice production are China (140 million tons), India (110 million tons), which makes up almost 60% of the global volume. They are followed by Thailand, the Philippines, Brazil, Japan, and Vietnam with lower productivity [4–5]. The total rice production in Ukraine reaches 100 thousand tons/year with 20 thousand tons of rice husk. The use of rice husk allows expanding the raw material resource base, creating an effective, environmentally friendly production for extraction of amorphous silicon(IV) oxide that meets the specified requirements. RH mostly consists of lignin (according to [6–9] ~ 40% mass), cellulose (according to 36% mass) and around 24% mass of silicon(IV) oxide and metal oxides [9; 10]. RH can be used as raw material for amorphous silicon(IV) oxide extraction for various purposes. The advantage of this raw material is its yearly reproducibility and possibility to get valuable components with pre-specified properties [9; 11–16].

The traditional methods of obtaining silicon(IV) oxide from mineral raw materials are multistage and energy-consuming. The extraction

of amorphous silicon(IV) oxide from RH reduces energy consumption, and the resulting product has a purity of 99.9 % of the mass. A number of papers [17–23] include the results of experiments on rice husk chemical processing in various process modes. To obtain high purity amorphous silicon dioxide, it is necessary to extract carbon-containing compounds from rice husk.

Thermodynamic research [8] made it possible to determine the operating parameters of the RH heat treatment process (temperature, pressure, the composition of the gaseous phase), which allowed reducing the amount of experimental studies and planning them. However, thermodynamic studies do not allow to determine the temporal characteristics of the process. The purpose of this paper is to study the laws of thermal decomposition of rice husks freed from lignin. Determination of the kinetic parameters of this process in non-isothermal conditions, the establishment of the mechanism of the process of thermal decomposition. Compilation of a mathematical model that fully describes this process.

Experimental

A derivatographic method is used when studying the kinetics of thermal decomposition of rice husks freed from lignin [24; 25]. This method allows applying a complex procedure of concurrent analysis of mass variation curves (TG), and temperature change (DTA). The derivatographic method in line with other methods of the analysis (chemical, and X-ray phase, etc.) allows determining the chemistry of transformations at samples heating and

calculating the reaction activation energy E and the pre-exponential factor k_0 . A sample of rice husk was subjected to a derivatographic study, from which lignin was extracted with an alcoholic acid extractant according to the procedure [27]. The composition of the residue after the

extraction of lignin and decomposition products during thermal decomposition was determined by size exclusion chromatography using a CHNS / O 2400 SeriesII analyzer. The obtained results are shown in table 1.

Table 1

The elemental composition and ash content of rice husk freed from lignin

Parameter	Symbol, UOM	Actual Value
Ash content, dry basis	A ^d ,%	31.8
Carbon Mass Content	C ^d ,%	23.52
Hydrogen Mass Content	H ^d ,%	4.694
Nitrogen Mass Content	N ^d ,%	0.28
Oxygen Mass Content	O ^d ,%	39.76

Based on the data given in table 1, a conditional formula of a rice husk molecule, freed from lignin, of the following form was proposed: $C_{1,96}H_{4,64}O_{2,48}N_{0,02} \cdot 0.53SiO_2 \cdot 0.30H_2O$.

A derivatogram was obtained using a Paulik F.- Paulik J.- Erdey L. system derivatograph in air in the temperature range 20–1000 °C at a heating rate of 0.17 K/s. Statistical processing of the obtained results and mathematical modeling were carried out using standard Mathcad 14 software.

Results and discussion

Fig. 1 shows a derivatogram of RH sample thermal decomposition after the RH lignin removal in air. As one can see in Fig. 1, three peaks are recorded on the thermal image of the sample. The first endoeffect is observed at 102 °C, and two exoeffects correspond to 355 °C and 600 °C respectively. The main organic components of rice husk are cellulose and lignin with burn temperature of 350 °C and 500 °C respectively [28–30]. Therefore, we can make a conclusion that the exoeffects on the derivatogram correspond to the burning of cellulose and some lignin remained unextracted from RH.

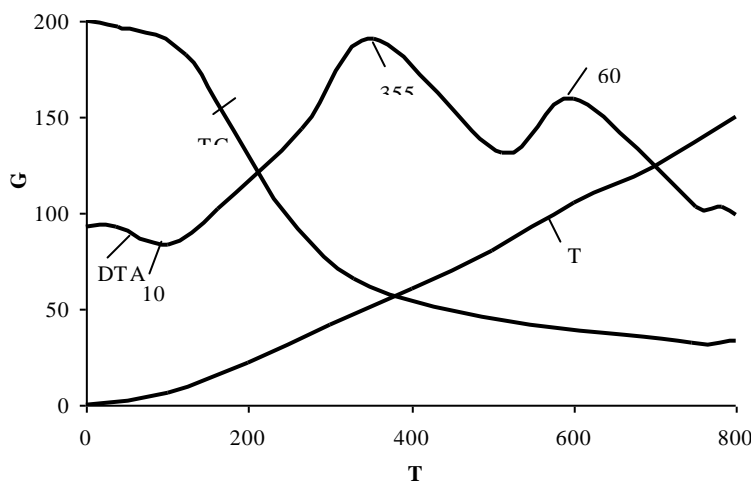
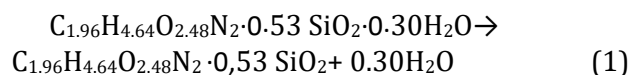
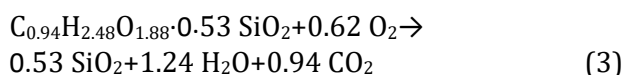
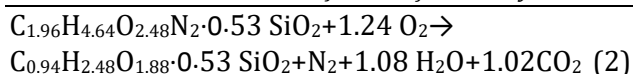


Fig.1. Derivatogram of the thermal decomposition process of a sample of rice husk freed from lignin in air

At 102 °C water is evaporated. Based on the change in the sample mass, the conditional initial rice husk molecule freed from lignin, $C_{1,96}H_{4,64}O_{2,48}N_{0,02} \cdot 0.53SiO_2 \cdot 0.30H_2O$, can be divided into its dehydrated part and water. At 250–530 °C, cellulose thermal oxidative degradation occurs along with formation of product $C_{0,94}H_{2,48}O_{1,88} \cdot 0.53SiO_2$. Then at 530–730

°C, the further thermal degradation of residual carbon-containing components occurs along with silicon(IV) oxide formation. Thus, we can write the following scheme of chemical transformations:





According to the proposed scheme of decomposition of rice husk free from lignin, with the release of silicon dioxide during chemical transformations, seven components are involved.

We designate them with symbols, as shown in Table 2.

Table 2

Component Symbols	
Component	Symbols
$C_{1.96}H_{4.64}O_{2.48}N_2 \cdot 0.53 SiO_2 \cdot 0.30 H_2O$	n_1
$C_{1.96}H_{4.64}O_{2.48}N_2 \cdot 0.53 SiO_2$	n_2
$C_{0.94}H_{2.48}O_{1.88} \cdot 0.53 SiO_2$	n_3
N_2	n_4
H_2O	n_5
CO_2	n_6
SiO_2	n_7

The mathematical model that corresponds to this process consists of three differential kinetic and four algebraic equations of material balance:

$$\frac{dn_1}{d\tau} = -k_1 n_1 \quad (4)$$

$$\frac{dn_2}{d\tau} = k_1 n_1 - k_2 n_2, \quad (5)$$

$$\frac{dn_3}{d\tau} = k_2 n_2 - k_3 n_3 \quad (6)$$

$$n_4 = 0,01n_1^0 - 0,01n_1 - 0,01n_2 \quad (7)$$

$$n_5 = 2,62n_1^0 - 2,62n_1 - 2,32n_2 - 1,24n_3 \quad (8)$$

$$n_6 = 1,96n_1^0 - 1,96n_1 - 1,96n_2 - 0,94n_3 \quad (9)$$

$$n_7 = 0,53(n_1^0 - n_1 - n_2 - n_3) \quad (10),$$

where $k_1 - k_3$ - are the reaction rate constants (1-3), s^{-1} ;

τ - time, s;

n_1^0 - the initial number of moles of rice husk freed from lignin;

$n_1 - n_7$ - the current number of moles of the corresponding components.

To determine the kinetic parameters of the RH thermal decomposition process after the lignin removal, peaks in the thermal image (Fig. 1) that correspond to reactions (1-3) are determined. The reference temperatures are determined by the bend point on the DTG curve and are as follows: $T_{s1} = 375$ K, $T_{s2} = 628$ K, $T_{s3} = 823$ K.

Arrhenius equation is used to define the specific reaction rate:

$$k = k_0 \exp\left(-\frac{E}{RT}\right) \quad (11),$$

where k - specific reaction rate, c^{-1} ;

k_0 - pre-exponential factor, c^{-1} ;

T - varying temperature, K.

Relative activation energy is defined using the following equation:

$$E = tg\beta \cdot RT_s^2 \quad (12),$$

where T_s - reference temperature, K;

$tg\beta$ - slope of the line made in $W^* = f(\theta)$ coordinates.

Equation [31] is used to plot $W^* = \ln \ln \frac{W_0 - W_k}{W_0 - W_\tau}$

against temperature θ :

$$\ln \ln \frac{W_0 - W_k}{W_0 - W_\tau} = \frac{E \cdot \theta}{RT_s^2} \quad (13),$$

where W_0 , W_τ , W_k are the initial, current and final sample mass respectively, g;

E - relative reaction activation energy, J/mol;

R - universal gas constant, $R = 8.310$ J/(mol·K);

θ - temperature difference, taken with an arbitrary step on the derivatogram. For calculations, we accept $\theta_1 = \pm 10$ K, $\theta_2 = \pm 20$ K, $\theta_3 = \pm 30$ K

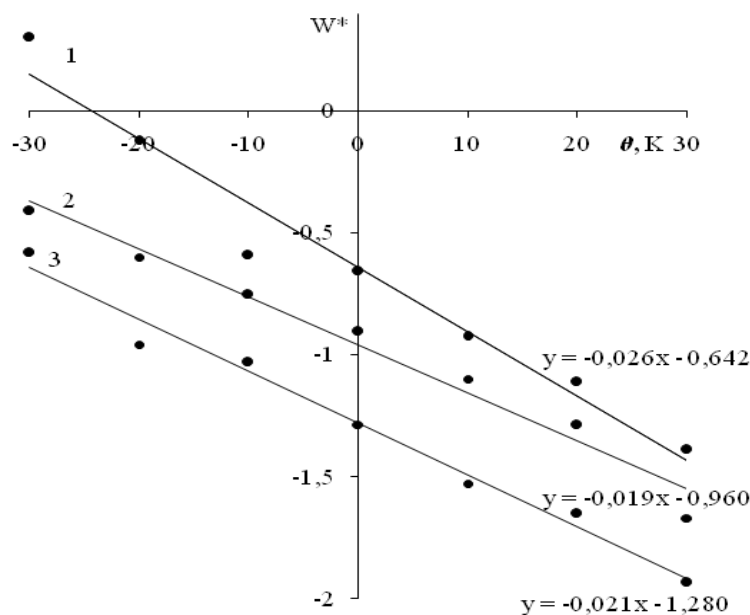


Fig.2. Dependences of W^* on the temperature difference θ for reactions (1-3): 1 – for reaction (1) at a temperature $T_{s1} = 375$ K; 2– for reaction (2) at a temperature $T_{s2} = 628$ K; 3– for reaction (3) at a temperature $T_{s3} = 823$ K.

Equation [24; 31] is used to define pre-exponential factor k_0 :

$$k_0 = \frac{g \cdot E}{RT_s^2 \exp\left(-\frac{E}{RT_s}\right)} \quad (14),$$

where g – sample heating rate, K/sec.

The calculated values of E and k_0 are given in Table 3.

Kinetic parameters of the process of thermal decomposition of lignin-free rice husk

Table 3

Chemical Reaction	T, K	Pre-Exponential Factor, k_0 , c-1	Relative Reaction Activation Energy, E, J/mol
1	375	$0.69 \cdot 10^2$	$30.8 \cdot 10^3$
2	628	$1.23 \cdot 10^4$	$62.95 \cdot 10^3$
3	823	$1.00 \cdot 10^5$	$119 \cdot 10^3$

When solving the system of equations (4–10) using standard Mathcad 14 software, the spatiotemporal properties of the process of thermal decomposition of lignin-free rice husk are determined (Fig. 3).

The proposed mathematical model allows us to calculate the degree of conversion of lignin-

free rice husk to silicon (IV) oxide, depending on the temperature of thermal decomposition. The dependence obtained as a result of solving the system of equations (4–10) is shown in Fig. 4 by a solid line.

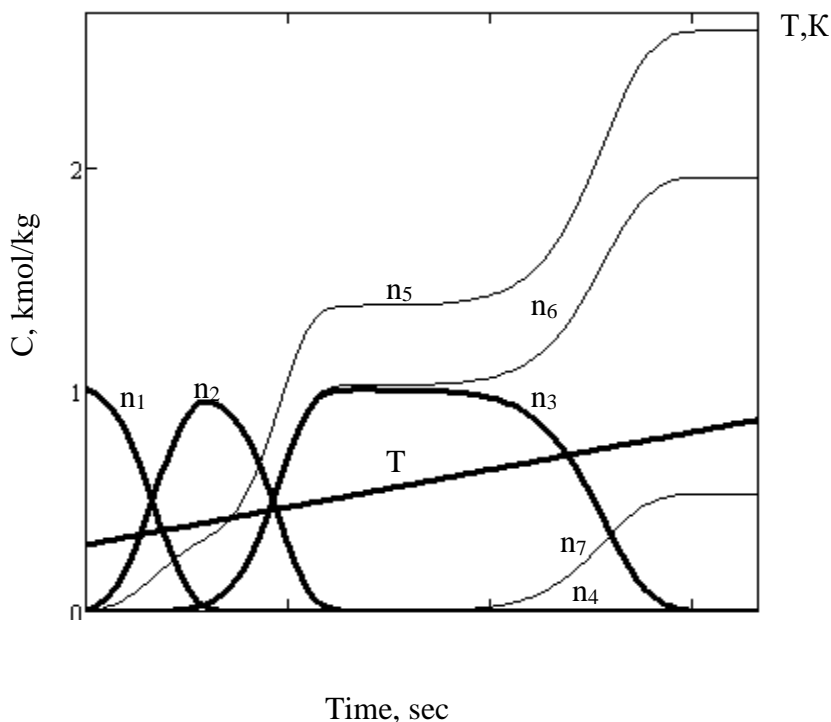
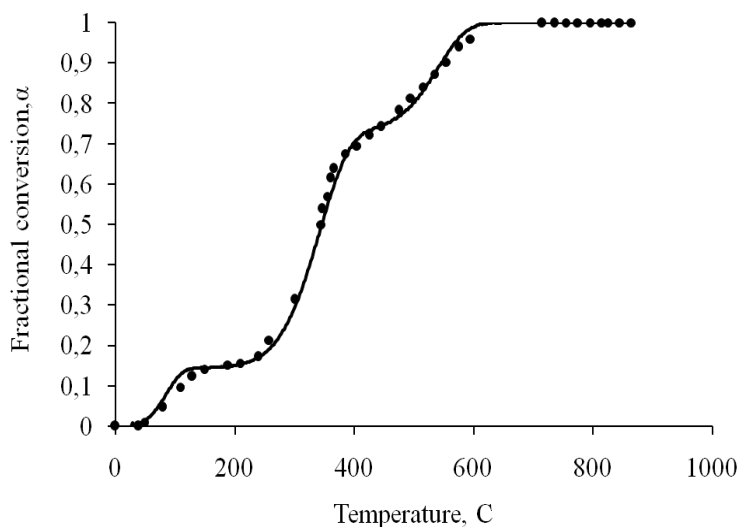


Fig. 3. The ratio of the concentrations of reaction products in the process of thermal decomposition of lignin-free rice husk



— is the calculated curve;
● - experimental values.

Fig. 4. Temperature dependence of the conversion of lignin-free rice husk to silicon(IV) oxide on temperature

The calculation of the degree of conversion on the basis of experimental data was carried out according to the formula:

$$\alpha = \frac{W_0 - W_s}{W_0 - W_K} \quad (15)$$

where W_0 - is the initial mass of the sample;
 W_s - is the mass of the sample at TS;
 W_K - is the final mass of the sample.

The values of the degree of conversion calculated from the experimental data are shown in Fig. 4 by dots.

The proposed mathematical model allows establishing the dependence of the time of formation of silicon(IV) oxide with a degree of conversion equal to unity on the heating rate of rice husk free of lignin. As can be seen from Fig. 5, the minimum time spent on this process with a degree of conversion equal to unity corresponds

to a heating rate of 0.6 K/s. The calculated value is in good agreement with the experimental data.

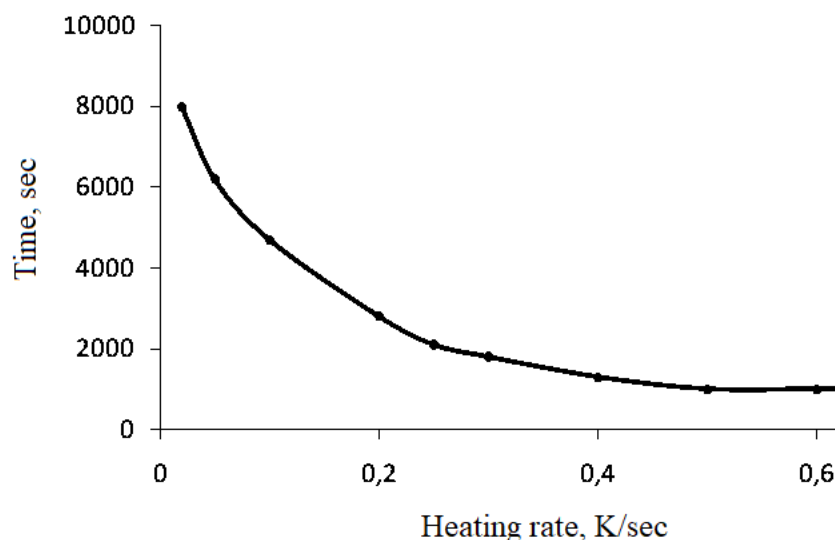


Fig. 5. Dependence of the time for the release of silicon(IV) oxide on the heating rate of a sample of lignin-free rice husk

Consequently, the proposed mathematical model adequately describes the experimental data and can be used in the design of reactors for the processing of lignin-free rice husks in the production of high-purity amorphous silicon(IV) oxide.

Conclusions:

1. Based on the obtained experimental data on the kinetics of thermal decomposition of lignin-free rice husk, the scheme of this process has been established. It has been shown that the formation of silicon(IV) oxide occurs during successive conversions of the conditional molecules $C_{1.96}H_{4.64}O_{2.48}N_{2 \cdot 0.53}SiO_2 \cdot 0.30H_2O$, $C_{1.96}H_{4.64}O_{2.48}N_{2 \cdot 0.53}SiO_2$, $C_{0.94}H_{2.48}O_{1.88} \cdot 0.53 SiO_2$ at temperatures of 102, 355 and 600 °C.

2. A mathematical model for description of the kinetics of the thermal decomposition of lignin-free rice husks has been developed. The kinetic parameters of the process were obtained and an experimental verification of the adequacy of the proposed model was carried out.

3. It has been shown that the formation of silicon (IV) oxide with a degree of conversion equal to unity occurs at a temperature of 600 °C. A minimum process time of 2000 seconds is provided at a heating rate of at least 0.6 K/s. It is proposed to use the developed mathematical model and the results of experimental studies in the design of reactors for processing lignin-free rice husks in the production of high-purity amorphous silicon(IV) oxide.

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