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INVESTIGATION OF THE THERMAL TRANSFORMATIONS OF RICE HUSK BY DIFFERENTIAL THERMAL ANALYSIS FOR THE PRODUCTION OF SILICON CARBIDE

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Abstract — The thermal transformations of rice husk obtained from rice cultivated in the Kyzylorda Region were investigated to determine the appropriate conditions for its preliminary thermal treatment prior to silicon carbide production. The study was conducted using differential thermal analysis (DTA) with a high-temperature simultaneous thermal analyzer STA300. The main temperature ranges of the thermal transformations of rice husk were determined. The DTA curve revealed an endothermic effect with a maximum at 150 °C, associated with moisture removal, as well as exothermic effects in the range of 300-440 °C and at higher temperatures, which were attributed to the sequential removal and transformation of organic components. It was established that the main mass loss occurred in the temperature range of 300-600 °C. Based on the results of thermoxidative treatment, the maximum removal of volatile components was achieved at 600-700 °C with a treatment duration of 10-20 min. The obtained results provide a basis for selecting the temperature conditions for the preliminary preparation of rice husk and can be used in the development of a technology for its further application as a silicon- and carbon-containing raw material for silicon carbide production.

Keywords — rice husk, thermal analysis, differential thermal analysis, DTA, thermal transformations, pyrolysis, thermoxidative treatment, volatile components, silicon carbide.

КРЕМНИЙ КАРБИДІН АЛУ ҮШІН КҮРІШ ҚАУЫЗЫНЫҢ ДИФФЕРЕНЦИАЛДЫ-ТЕРМИЯЛЫҚ ТАЛДАУ ӘДІСІМЕН ТЕРМИЯЛЫҚ ТҮРЛЕНУЛЕРІН ЗЕРТТЕУ

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Аңдатпа — Зерттеу жұмысында Қызылорда облысында өсірілген күріштен алынған күріш қауызының термиялық түрленулері зерттеліп, оны кремний карбидін алу алдында алдын ала термиялық өңдеудің тиімді жағдайлары анықталды. Зерттеу жоғары температуралы STA300 бір мезгілдегі термиялық анализаторын қолдана отырып, дифференциалды-термиялық талдау (ДТА) әдісімен жүргізілді. Күріш қауызының негізгі термиялық түрлену процестері жүретін температуралық аралықтар анықталды. ДТА қисығында 150 °C температурада максимумға жететін, ылғалдың жойылуымен байланысты эндотермиялық әсер, сондай-ақ 300-440 °C және жоғары температуралар аймағында органикалық компоненттердің біртіндеп жойылуы мен түрленуіне байланысты экзотермиялық әсерлер анықталды. Негізгі масса жоғалуы 300-600 °C температура аралығында жүретіні белгіленді. Термототықтырғыш өңдеу нәтижелері бойынша ұшпа компоненттердің барынша жойылуы 600-700 °C температурада 10-20 минут өңдеу ұзақтығында қамтамасыз етілетіні анықталды. Алынған нәтижелер күріш қауызына алдын ала өңдеу жүргізудің температуралық режимдерін негіздеуге мүмкіндік береді және оны кремний карбидін алу үшін құрамында кремний мен көміртегі бар шикізат ретінде одан әрі пайдалану технологиясын әзірлеуде қолданылуы мүмкін.

Түйін сөздер — күріш қауызы, термиялық талдау, дифференциалды-термиялық талдау, ДТА, термиялық түрленулер, пиролиз, термототықтырғыш өңдеу, ұшына компоненттер, кремний карбиді.

ИССЛЕДОВАНИЕ ТЕРМИЧЕСКИХ ПРЕВРАЩЕНИЙ РИСОВОЙ ШЕЛУХИ МЕТОДОМ ДИФФЕРЕНЦИАЛЬНО-ТЕРМИЧЕСКОГО АНАЛИЗА ДЛЯ ПОЛУЧЕНИЯ КАРБИДА КРЕМНИЯ

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Аннотация — В работе исследованы термические превращения рисовой шелухи, полученной из риса, выращенного в Кызылординской области, с целью определения рациональных условий ее предварительной термической обработки для последующего получения карбида кремния. Исследование проводили методом дифференциально-термического анализа с использованием высокотемпературного одновременного термического анализатора STA300. Установлены основные температурные интервалы протекания термических превращений рисовой шелухи. На ДТА-кривой выявлен эндотермический эффект с максимумом при 150 °С, связанный с удалением влаги, а также экзотермические эффекты в области 300-440 °С и при повышенных температурах, обусловленные последовательным удалением и превращением органических компонентов. Установлено, что основная потеря массы происходит в температурном диапазоне 300-600 °С. По результатам термоокислительной обработки определено, что максимальное удаление летучих компонентов достигается при температуре 600-700 °С и продолжительности обработки 10-20 мин. Полученные результаты позволяют обосновать температурный режим предварительной подготовки рисовой шелухи и могут быть использованы при разработке технологии ее дальнейшего применения в качестве кремний- и углеродсодержащего сырья для получения карбида кремния.

Ключевые слова — рисовая шелуха, термический анализ, дифференциально-термический анализ, ДТА, термические превращения, пиролиз, термоокислительная обработка, летучие компоненты, карбид кремния.

KEY FINDINGS

The thermal transformations of rice husk were investigated by differential thermal analysis using a STA300 analyzer. The main temperature ranges of thermal effects and mass loss were established, and the conditions for thermoxidative treatment ensuring maximum removal of moisture and volatile components at 600-700 °C for 10-20 min were determined. The obtained results can be used to justify the preliminary preparation of rice husk as a silicon- and carbon-containing raw material for silicon carbide production.

INTRODUCTION

Silicon carbide (SiC), owing to its unique properties, including high hardness and refractoriness, is widely used in electrical engineering, semiconductor, radio engineering, tooling, and other industrial applications. Existing industrial methods for producing silicon carbide from mineral raw materials are highly energy-intensive and costly, while the resulting silicon carbide does not always meet the required quality standards [1].

The Acheson process [2] is an industrial method for producing silicon carbide. The synthesized silicon carbide obtained by this method is used in the manufacture of abrasive tools, as a vapor source for crystal growth by the Lely method, and for the production of silicon carbide-based products by pressing and slip casting. According to this method, low-ash coke or petroleum coke is added to silicon dioxide, followed by heat treatment in which an electric current is passed through carbon electrodes and the core at a temperature of 2500–2600 °C. The process duration is approximately 40 h. Green or black polycrystalline silicon carbide is also added to the charge.

Until the early 1980s, the Lely method [3] was the only industrial method for producing silicon carbide crystals for the semiconductor industry. Even today, SiCrystal (Germany) uses the Lely method to produce polished 6H- and 15R-SiC crystals. The Lely method is based on the evaporation of polycrystalline silicon carbide at temperatures of 2500–2650 °C, followed by vapor condensation on randomly formed nuclei.

The spontaneous nucleation characteristic of the Lely method results in the uncontrolled formation of

a large number of small crystals. The use of seed crystals eliminates these limitations and enables the growth of large-diameter bulk single crystals. Currently, this approach is known as the Physical Vapor Transport (PVT) method. The method is based on the condensation of a supersaturated vapor generated by the sublimation of synthesized silicon carbide onto a single-crystal seed.

Industrial silicon carbide is produced in two main varieties, distinguished by color: green and black. They differ relatively little in chemical composition and abrasive properties; however, green silicon carbide contains fewer impurities and is considered a higher-quality abrasive than black silicon carbide. The production of silicon carbide by carbothermic reduction [4] can be represented by the following equation:



The formation of silicon carbide involves the interaction of silicon dioxide with carbon at a temperature of 2200-2300 °C. In the present study, the production of silicon carbide from rice husk is considered. Rice husk obtained from rice cultivated in the Kyzylorda Region was used as the raw material. To determine the main physicochemical characteristics of the pyrolyzed rice husk, the samples were subjected to thermal analysis using differential thermal analysis (DTA).

Research Gap. Although rice husk has been widely studied as a renewable silicon- and carbon-containing raw material, insufficient attention has been given to the detailed characterization of its thermal transformations under controlled heating conditions for subsequent silicon carbide production. In particular, the temperature ranges of the main thermal effects, the stages of moisture and volatile component removal, and the relationship between thermal behavior and the conditions of preliminary thermoxidative treatment require further clarification. Therefore, a detailed DTA-based study of rice husk is necessary to establish rational conditions for its thermal preparation prior to silicon carbide production.

Scientific Novelty. The scientific novelty of this study lies in the identification of the main thermal transformation stages of rice husk using differential thermal analysis (DTA) and the determination of the corresponding temperature ranges. The temperature conditions and duration of thermoxidative treatment required for the effective

removal of moisture and volatile components were established. The obtained results provide a scientific basis for the preliminary thermal preparation of rice husk as a silicon- and carbon-containing raw material for subsequent silicon carbide production.

MATERIALS AND METHODS

Differential thermal analysis (DTA) is a thermal analysis technique used to investigate thermal effects occurring in a material during heating or cooling. The method is based on the simultaneous measurement of the temperatures of the sample and an inert reference material under a controlled temperature program and determination of the temperature difference between them:

$$\Delta T = T_s - T_r \quad (2)$$

where T_s is the temperature of the sample and T_r is the temperature of the reference material.

In the absence of thermal effects, the temperatures of the sample and reference material change in a similar manner. When a physical or chemical transformation occurs in the sample, a temperature difference develops between the sample and the reference material, which is recorded as a peak on the DTA curve.

Differential thermal analysis was performed using a high-temperature simultaneous thermal analyzer STA300 (Figure 1). The method is based on recording the temperature difference between the investigated sample and the inert reference material under a controlled temperature program. During heating, changes in the thermal behavior of the sample associated with physical and chemical transformations were recorded.

The analysis was performed over a temperature range from room temperature to 1250 °C. The heating rate was set within the range of 1–80 °C/min, depending on the experimental conditions. The instrument is equipped with a controlled gas supply system that allows measurements to be performed under inert, oxidizing, or reducing atmospheres, as well as under static or dynamic conditions. The temperature resolution of the instrument is 0.1 °C, while the DTA/DSC measurement range is 0 to ±500 mW.

The obtained thermal curves were used to determine the onset, peak, and endset temperatures of endothermic and exothermic effects, as well as to establish the temperature ranges in which the corresponding physicochemical transformations occurred.

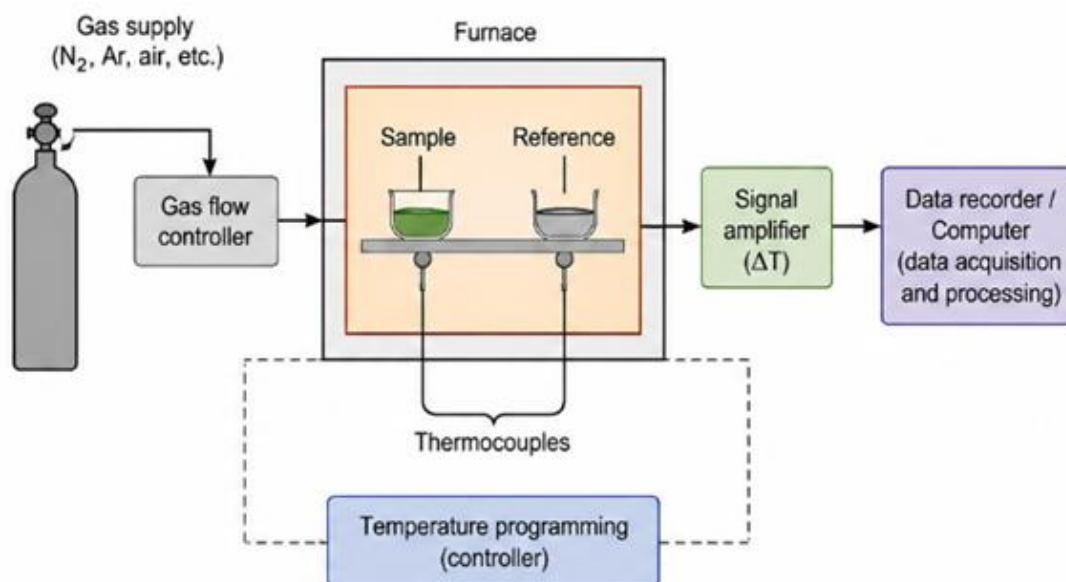


Figure 1 – Schematic diagram of the DTA equipment

RESULTS AND DISCUSSION

Within the investigated temperature range, the DTA curve (Figure 2) shows a pronounced endothermic effect with a maximum at 150 °C, which is associated with moisture evaporation. An exothermic effect at 300 °C is also observed,

indicating the onset of volatile component release and continuing through the temperature range of 390, 440, and 790 °C. The mass loss associated with moisture removal and the release of volatile components amounted to 7.5-10% and 50-60%, respectively.

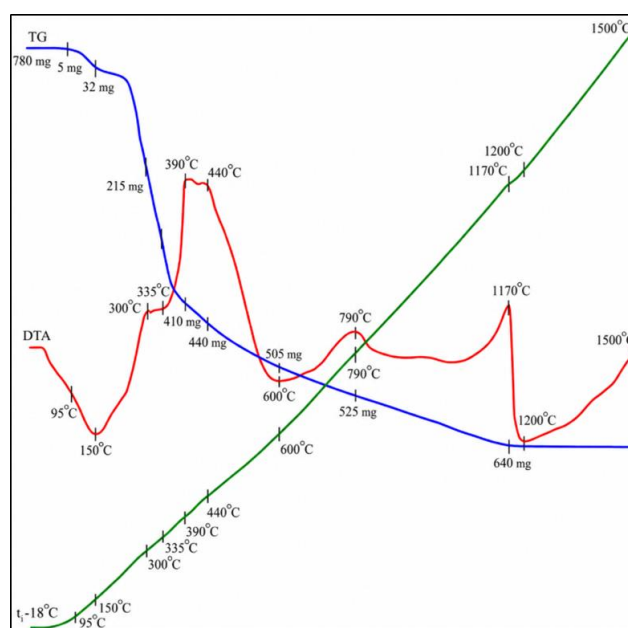


Figure 2 – DTA curve of the thermal transformations of rice husk

Thermoxidative treatment (pyrolysis) of rice husk (RH) was investigated to determine the mass loss associated with moisture and volatile organic components during heat treatment in the temperature

range of 150–700 °C, at atmospheric pressure, and for treatment durations of 10–60 min. It was established that the mass loss of RH at 150, 200, 300, 400, 500, 600, and 700 °C was 7.02 and 11.88%, 7.07 and 10.53%, 23.92 and 43.46%, 52.32 and 52.74%, 57.32

and 61.20%, 62.38 and 62.34%, and 62.20 and 61.84%, respectively, for treatment durations of 10 and 60 min. Therefore, to achieve maximum removal of volatile components from RH, thermoxidative treatment should be carried out in the temperature range of 600-700 °C for 10-20 min.

CONCLUSIONS

1. Differential thermal analysis of pyrolyzed rice husk was performed using a high-temperature simultaneous thermal analyzer STA300. The main temperature ranges of thermal transformations and the corresponding endothermic and exothermic effects were determined.

2. A pronounced endothermic effect with a maximum at 150 °C was identified on the DTA curve, primarily associated with moisture removal. With further heating, exothermic effects were observed in the range of 300-440 °C, as well as at higher temperatures around 790 °C, indicating successive thermal transformations of the organic components of rice husk.

3. It was established that the main mass loss of rice husk occurs in the temperature range of 300-600 °C. During thermoxidative treatment, the mass loss increased with increasing temperature and reached maximum values at 600-700 °C, after which further temperature increases resulted in only minor changes in mass.

4. Based on the results of thermoxidative treatment, a temperature range of 600-700 °C with a treatment duration of 10-20 min was identified as

optimal for the maximum removal of moisture and volatile organic components from rice husk. The obtained results can be used to substantiate the conditions for the preliminary thermal treatment of rice husk prior to its further use as a silicon- and carbon-containing raw material..

AUTHOR CONTRIBUTION

The author independently performed the formulation of the research objectives, experimental studies, processing and analysis of the obtained results, and preparation of the manuscript.

CONFLICT OF INTERESTS

The author declares no conflict of interest.

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СПИСОК ЛИТЕРАТУРЫ

- [1] Г. Г. Гнесин, Карбидокремниевые материалы. Москва, Россия: Металлургия, 1977. – 216 с.
- [2] О. А. Агеев, А. Е. Беляев, Н. С. Болтовец и др., Карбид кремния: технология, свойства, применение. Харьков, Украина: ИСМА, 2010. – 532 с.
- [3] Л. Я. Марковский, Д. Л. Оршанский, В. П. Прянишников, Химическая электротермия. Ленинград, СССР: Государственное научно-техническое издательство химической литературы, 1952. – 408 с.
- [4] И. С. Кайнарский, Э. В. Дегтерева, Карборундовые огнеупоры. Москва, Россия: Металлургиздат, 1963. – С. 9–14..

REFERENCES

- [1] G. G. Gnesin, Carbide-Silicon Materials. Moscow, Russia: Metallurgy, 1977, 216 p.
- [2] O. A. Ageev, A. E. Belyaev, N. S. Boltovets, et al., Silicon Carbide: Technology, Properties, Applications. Kharkov, Ukraine: ISMA, 2010, 532 p.
- [3] L. Ya. Markovsky, D. L. Orshansky, and V. P. Pryanishnikov, Chemical Electrothermy. Leningrad, USSR: State Scientific and Technical Publishing House of Chemical Literature, 1952, 408 p.
- [4] I. S. Kaynarsky and E. V. Degtereva, “Carborundum Refractories,” Moscow, Russia: Metallurgizdat, 1963, pp. 9–14.