

Article

Effect of Carbonization Temperature of Rice Husk Char Preparation on SiC Structure and Composition of By-Products in SiC Synthesis with Magnesiothermic Reduction Method

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Abstract: The structure of rice husk char is one of the factors that influence the morphology of SiC and the composition of by-products in the magnesiothermic reduction. This structure can be improved by carbonization. Therefore, this study aims to examine the effect of the carbonization temperature of rice husk char on the structure of SiC and identify the by-products formed by the magnesiothermic reduction. The rice husk char was made by carbonization at various temperatures of 700, 800, 900, and 1000 °C. They were converted to SiC by magnesiothermic reduction at 600 °C, washing with hydrochloric acid, calcination at 700 °C in an air atmosphere, and washing with a mixture of hydrofluoric acid and acetic acid. The products were characterized by FT-IR, XRD, and SEM-EDS. The results showed that creating SiC using the magnesiothermic method with carbonization at a temperature of 1000 °C produced a polytype 2H-SiC, and the by-products were MgO, Mg₂SiO₄, and Si. MgO was successfully separated in the washing step with a hydrochloric acid solution, while Mg₂SiO₄ was separated from SiC by washing using a mixture of hydrofluoric acid and acetic acid.

Keywords: rice husk char; carbonization; magnesiothermic reduction; silicon carbide polytype



Citation: Solihudin, S.; Haryono, H.; Noviyanti, A.R.; Umar, A.A. Effect of Carbonization Temperature of Rice Husk Char Preparation on SiC Structure and Composition of By-Products in SiC Synthesis with Magnesiothermic Reduction Method. *Processes* **2023**, *11*, 133. <https://doi.org/10.3390/pr11010133>

Academic Editor: Pavel Mokrejš

Received: 16 November 2022

Revised: 21 December 2022

Accepted: 28 December 2022

Published: 2 January 2023



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1. Introduction

Silicon carbide (SiC) is a material with superior properties such as a high hardness and melting point, resistance to corrosion and oxidation, high bandgap, as well as good chemical stability [1]. These superior properties facilitate the application of SiC in various fields such as ceramics, semiconductors, composites, solar cells, and photocatalysts. Meanwhile, its application depends on the type of structure and morphology [2].

SiC is generally synthesized with carbothermal reduction methods [1], sol-gel [3], chemical vapor deposition [4], and microwaves [5]. However, these methods have some drawbacks. The carbothermal reduction method requires a high temperature of about 1400 °C, the sol-gel method produces agglomerates in the SiC product, while the chemical vapor deposition and molten salt methods require special equipment.

An alternative method for the synthesis of SiC at a lower temperature of approximately 600 °C is magnesiothermic reduction (MR). In the MR method, SiC is formed through the reduction of SiO₂ by Mg to Si, which then reacts with C to form SiC [6]. Additionally, the manufacturing of SiC with the MR method also forms various by-products depending on the basic materials used. The manufacturing of SiC from TEOS (Tetraethyl ortosilicate, a synthetic source of silica) and C powder produces by-products in the form of MgC₂ or Mg₂C₃ [7]. Saeedifar et al. (2017) made SiC from an MCM-48/polyacrylamide composite with Si, Mg₂Si, and Mg₂SiO₄ as by-products [8]. Meanwhile, Su et al. (2016) found the formation of Mg₂Si by-products when SiC was synthesized from rice husk char [9].

Using a basic material for making SiC with the criteria of being environmentally friendly, abundant, and inexpensive has captured the attention of various investigations in the last decade. Rice husk char is a biomass waste that can be used as a basic material for making SiC because it contains C and SiO₂ [10]. The polytype is a reference in SiC applications in various fields. Therefore, the manufacturing of various SiC polytypes is the main attraction for various studies. Investigations on the manufacturing of SiC from rice husk char with the carbothermal reduction [11] and MR method [9] produced a 3C-SiC polytype. Meanwhile, the 2H-SiC polytype was successfully created by Gao et al. (2011) using the MR method with the SiO₂/C composite as the base material [12]. Gao et al. (2011) also stated that the structure and porosity of carbon affect the crystal structure of the SiC formed [12]. The structure and porosity of rice husk char can be changed through carbonization at high temperatures. Aside from porosity, the carbonization of rice husk char also causes the graphitization of C [13]. Therefore, this study aims to examine the effect of the carbonization temperature of the rice husk char on the SiC crystal structure and composition of by-products in the synthesis of SiC using the MR method.

2. Materials and Methods

2.1. Rice Husk Char Carbonization

Rice husk char from the gasification process was purified from metal oxide impurities by extraction with 1 M HCl (Sigma-Aldrich, Singapore) at the boiling temperature of the mixture for 4 h. The extracted mixture was filtered, and then the residue was rinsed with distilled water until the pH was neutral or chloride free. This was confirmed by testing with a AgNO₃ solution (Sigma-Aldrich), and then the residue was dried at 110 °C for 3 h.

The carbonization of the rice husk char was performed at various temperatures of 700, 800, 900, and 1000 °C with a heating rate of 5 °C/min for 3 h with argon gas flowing. Rice husk char from variations in the carbonization temperature were given the symbols C07, C08, C09, and C10, while those without carbonization were marked as C00. The effect of the variations in the temperature on the changes in the functional groups in the rice husk char before and after carbonization was characterized by FT-IR/Fourier Transform-Infrared (PerkinElmer Spectrum 100, Waltham, MA, USA).

2.2. Synthesis, Purification, and Characterization of Silicon Carbide

Rice husk char from each variation in carbonization temperature was added with magnesium powder (Merck, Singapore) with a mole ratio of SiO₂/Mg = 1.0:2.5. The mixture was ground with a ball mill (Retsch GmbH, PM 400 model, Haan, Germany) for 30 min at a rotational speed of 200 rpm and then added with latex and ethanol. The product was dried in a vacuum oven (Napco Vacuum Oven 5831, Amityville, NY, USA) until all the steam was lost. Afterward, the mixture (SiO₂ and Mg) was formed into pellets by applying pressure of up to 4 tons. The pellets were reduced in a furnace (Nabertherm, GmbH, Lilienthal, Germany) at a temperature of 700 °C for 3 h with flowing argon gas. The reduction product of the gasified rice husk char was depicted with the symbol C00R. Meanwhile, the reduction product after being carbonized at various temperatures of 700, 800, 900, and 1000 °C was given the symbols C07R, C08R, C09R, and C10R, respectively.

The reduction product was purified from MgO by heating in 1 M HCl followed by stirring. After filtering, the residue was washed with distilled water until it reached a neutral pH. The reduced products from various preparations of the rice husk char, after washing with a 1 M HCl solution, were given the symbols C07RH, C08RH, C09RH, and C10RH. The washings products were dried and then characterized with FT-IR and XRD, while the reduction product from the purification step was calcined at 700 °C for 1 h in an air atmosphere. After cooling, the calcined products, namely C07KU up to C10KU, were characterized by FT-IR and XRD.

The calcined product was washed with a mixture of 5% HF and 4.36 M CH₃COOH (Merck, Singapore) on a 1:9 volume ratio. Washing was carried out for 1 h by stirring and was assisted by heating, and then the mixture from the wash was filtered. The residue was

washed with distilled water until the pH was neutral, and it was dried in a vacuum oven. The products from this last stage were given the symbols C07SiC up to C10SiC and were characterized by FT-IR, XRD, and SEM-EDS.

3. Results

3.1. Rice Husk Char Carbonization

The carbonization of the rice husk char was carried out to remove volatile impurities and increase the degree of carbonization and the regularity of the carbon structure. A comparison of the functional groups of the rice husk char before and after carbonation at various temperatures is shown in Figure 1 and Table 1.

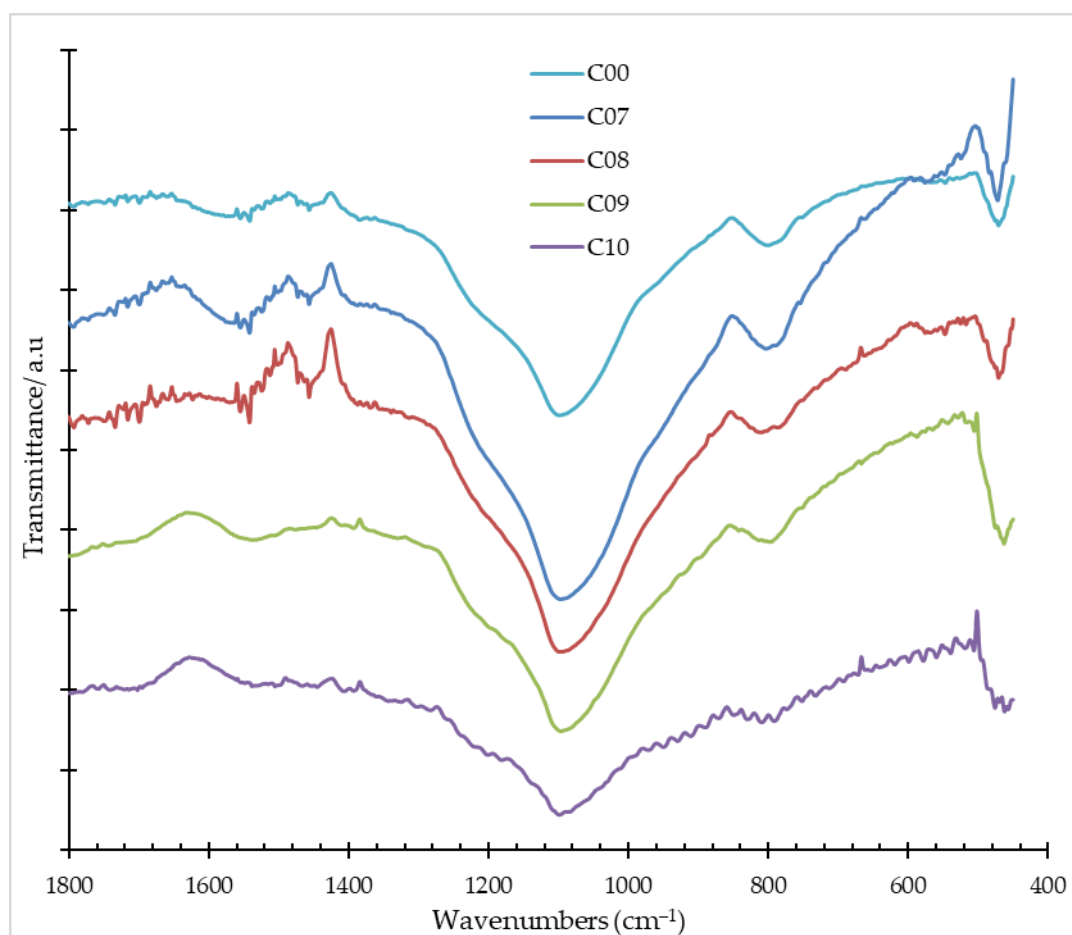


Figure 1. IR spectrum of rice husk char from gasification (C00) and after carbonization at temperature variations of 700, 800, 900, and 1000 °C (C07–C10).

Table 1. Comparison of IR absorption in rice husk char samples from gasification, carbonization with temperature variations, and standards.

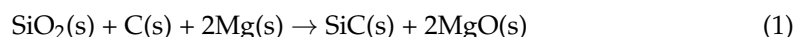
Type of Vibration	Wavenumber/cm ^{−1}					Standard [14]
	C00	C07	C08	C09	C10	
Bending vibration of ≡Si-O at (≡Si-O-Si≡)	472	472	471	463	-	465–475
Symmetric stretching vibration of =Si-O at (=Si-O-Si=)	799	803	810	799	-	800–870
Asymmetric stretching vibration of =Si-O at (=Si-O-Si=)	1099	1096	1095	1096	1099	1050–1150

Figure 1 shows a change in the IR absorption of the rice husk char before (C00) and after carbonization (C07–C10). The band around 1600 cm^{−1} is usually caused by the

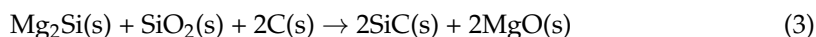
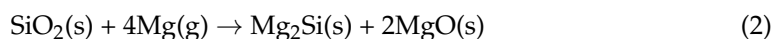
stretching vibration of C=O in ketones, aldehydes, lactones and carboxyl groups; the band around 1400–1500 cm^{−1} is attributed to an aromatic ring or C=C stretching vibration. This indicates the formation of carbonyl groups and the carbonization of the precursor. This shows that the degree of carbonization in C00 is smaller than that of rice husk char after carbonization. The relative intensity of the band around 1600 cm^{−1} displayed decreases with the change in the calcination temperature. For temperatures above 800 °C, the relative intensity of the band at 1600 cm^{−1} began to disappear with the increase in the activation temperature. The strong bands located around 1088 cm^{−1} and 803 cm^{−1} are attributed to the asymmetric and symmetric stretching of the Si–O band. The IR absorptions identified from all samples, as shown in Table 1, included the bending vibration of Si–O at Si–O–Si≡, symmetric stretching vibration of =Si–O at =Si–O–Si=, and asymmetric stretching vibration of =Si–O at (=Si–O–Si=). These absorption bands indicate the presence of silica in rice husk char.

3.2. SiC Synthesis and Purification with 1 M HCl Solution

SiC synthesis using the magnesiothermic reduction (MR) method was carried out by adding Mg as a reducing agent. Mg was used in a slightly higher amount than the stoichiometric requirement, namely the ratio of moles of SiO₂ to Mg of 1:2.5. The determination of the mole ratio refers to Equation (1) as proposed by Shi et al. [6]:



Su et al. (2016) confirmed that the reaction for the formation of SiC (Equation (1)) follows this mechanism: (a) SiO₂ reacts with Mg resulting in Mg₂Si, and (b) Mg₂Si and the remaining SiO₂ subsequently react with C, forming SiC and MgO complying with the Equations (2) and (3) [9]. The reaction between SiO₂ and Mg, according to Equation (2), is thermodynamically favorable due to a lower ΔG compared to the other possible reactions of the precursors, as can be seen from Equation (4) [6,15] and Equation (5) [7].



In this work, the MR method for the formation of SiC was accomplished at 700 °C. The ΔG of the reaction at a nonstandard condition can be calculated using Equation (6). According to the thermodynamics data table [16], the calculated ΔG from the chemical reactions in Equations (2), (4), and (5) at 700 °C (973.18 K) are −471.76, −327.88, and +77.86 kJ/mol, respectively. Meanwhile, the ΔG of reaction from Equation (3) is −401.44 kJ/mol; thus, the ΔG of the reaction in Equation (1) is −873.20 kJ/mol.

$$\Delta G = \Delta H^0 - T \Delta S^0 \quad (6)$$

The characterization of the functional groups by FT-IR for the reduction products of rice husk char variations (C07R–C10R) is shown in Figure 2 and Table 2. All samples of the reduction products had absorption at a wave number of about 830 cm^{−1}, which indicates the stretching vibration of Si–C. Additionally, all samples showed stretching vibrations of Mg–O at a wave number of 430 cm^{−1}. Among all the samples, C07R had a weak absorption at 430 cm^{−1}. Furthermore, it had an absorption of the asymmetric stretching vibration =Si–O on (=Si–O–Si=) at a wave number of 1093 cm^{−1}, indicating the presence of silica that has not reacted with magnesium which then forms SiC. This is because the C07 sample still contains several organic compounds that can react with Mg.

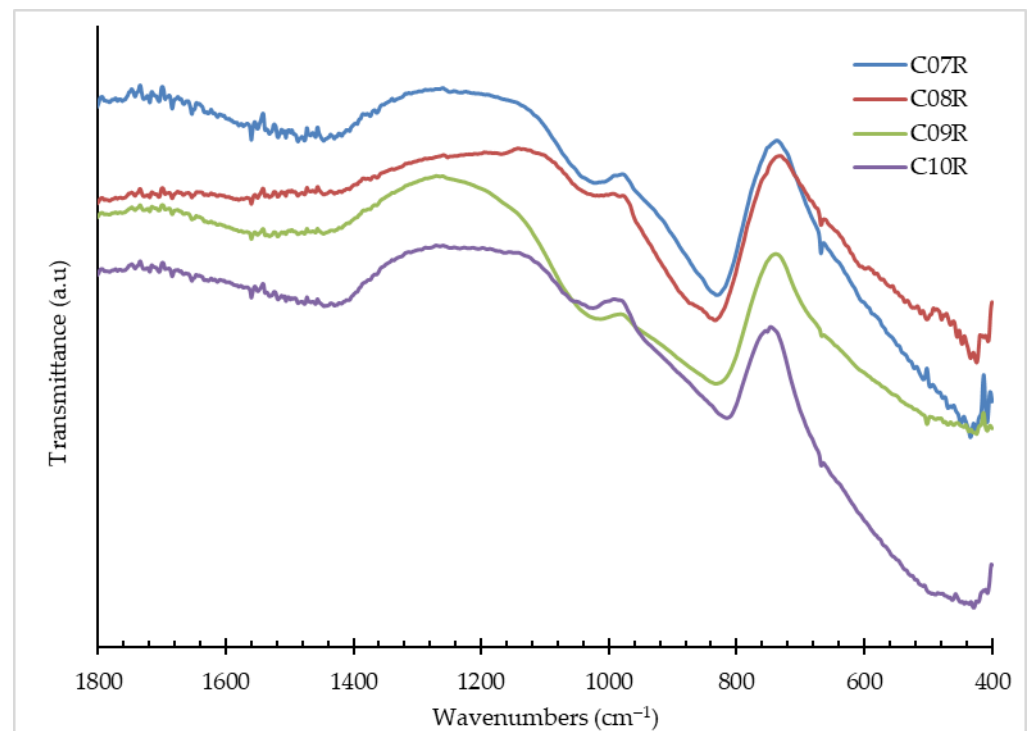


Figure 2. The IR spectrum of the reduction product with the basic ingredients of carbonization at temperatures of 700, 800, 900, and 1000 °C (C07R–C10R).

Table 2. Comparison of IR absorption on samples of reduction products from variations in the basic ingredients of rice husk char.

Type of Vibration	Wavenumber/cm ^{−1}				Standard
	C07R	C08R	C09R	C10R	
Stretching vibration of Mg–O	424	424	434	429	430 [11]
Stretching vibration of Si–C	834	832	831	815	830 [17]
Asymmetric stretching vibration of =Si–O at (=Si–O–Si=)	1093	1092	1091	1093	1050–1150 [14]

The XRD characterization results of each sample from the reduction stage are shown in Figure 3. The XRD pattern in Figure 3 shows that the four samples of the reduction products formed compounds with different types and compositions. Tracing the diffractogram with the Highscore Plus Ver program 2012.3.0.5 based on standard Inorganic Crystal Structure Database (ICSD) data, Figure 3 shows the formation of SiC at the peak at an angle of 2θ around 35.6° , 59.9° , and 71.7° (ICSD 98-016-4973). The by-products of the reduction step were MgO, Mg_2SiO_4 , and Si. The formation of MgO was indicated by a high-intensity peak at 2θ around 43.0° , 62.5° , and 78.9° (ICSD 98-010-4845), and it was formed in all types of reduction product samples. Other by-products were in very small quantities as demonstrated by a very low diffraction intensity. The by-product was estimated as Mg_2SiO_4 , as indicated by the presence of a diffraction peak at 2θ around 22.6° , 32.0° , 36.1° , and 51.7° (ICSD 98-003-4232). Meanwhile, silicon by-products were shown to peak at 2θ around 28.4° , 47.2° , and 56.1° (ICSD 96-901-3104). Sadique (2010) stated that the synthesis time and temperature played an important role in the formation of Mg_2SiO_4 [18]. Equation (7) is presumably the pathway for the formation of Mg_2SiO_4 [19]. The side reaction of Mg_2SiO_4 formation (Equation (7)) at 700 °C denotes a ΔG value of -62.65 kJ/mol. The value of ΔG from this side reaction is relatively much larger than the ΔG from the reaction for the

formation of SiC, so that in thermodynamic theory it will be formed in relatively small amounts. This fact is confirmed by the data from ref. [16].

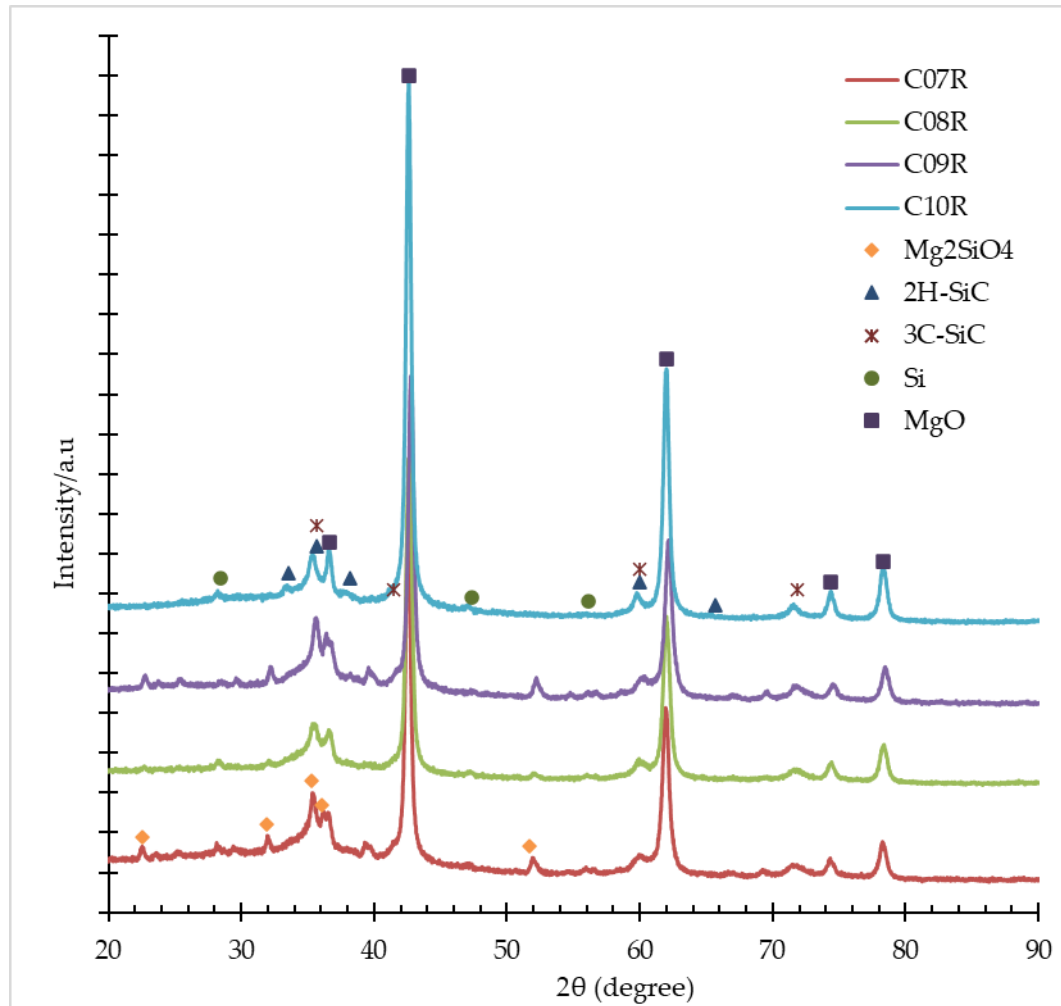
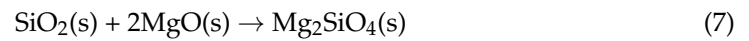


Figure 3. Diffraction pattern of reduction products from rice husk char base with various treatments.

The presence of MgO as a by-product in each type of reduction product was reduced by washing with a 1 M HCl solution. MgO reacts with HCl to form MgCl_2 , which is soluble in water. The FT-IR characterization of the reduction products after the washing step is shown in Figure 4 and Table 3. Based on the results, MgO was successfully washed from all samples, as indicated by the absence of the Mg-O vibrational absorption band at 430 cm^{-1} . It was also clarified by the absence of a diffraction peak at 2θ around 43.0° , 62.5° , and 78.9° on the C07H and C10H diffractograms.

Table 3. Comparison of absorption of product from washing with 1 M HCl.

Type of Vibration	Wavenumber/ cm^{-1}				
	C07RH	C08RH	C09RH	C10RH	Standard
Bending vibration of $\equiv\text{Si-O}$ at ($\equiv\text{Si-O-Si}\equiv$)	472	-	-	-	465–475 [14]
Stretching vibration of Si-C	829	829	835	815	830 [17]
Asymmetric stretching vibration of $=\text{Si-O}$ at ($=\text{Si-O-Si}=$)	1096	-	-	-	1050–1150 [14]

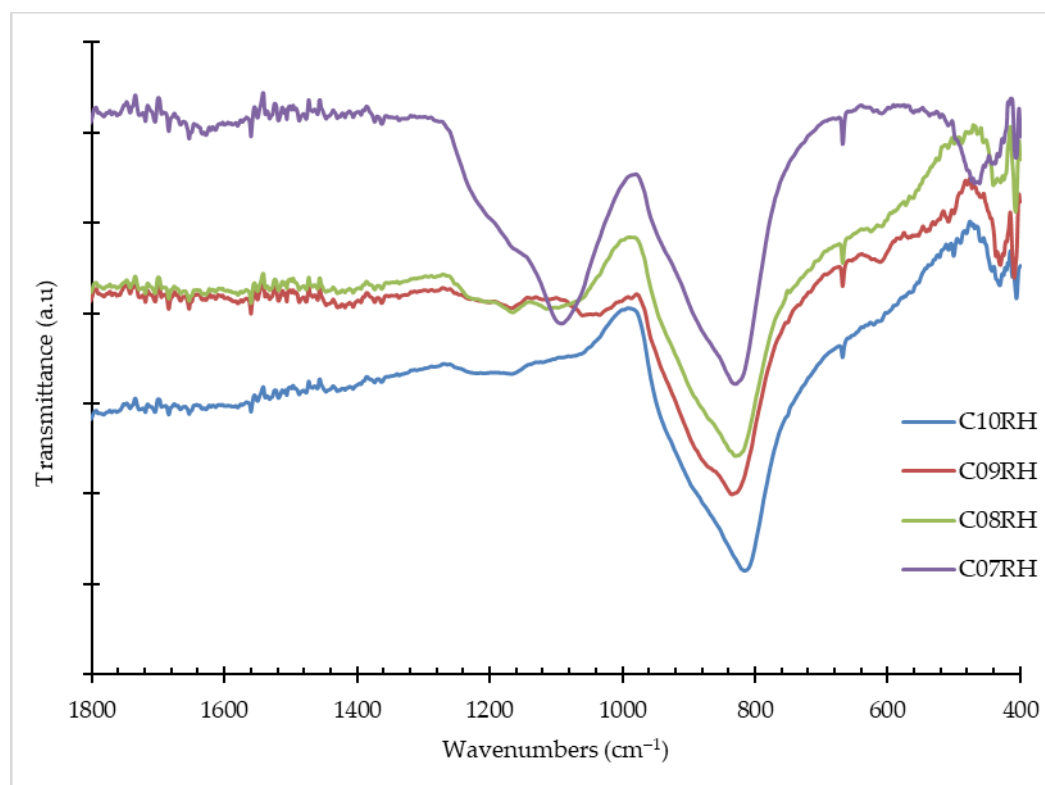


Figure 4. IR spectrum of the reduction product after washing with 1 M HCl solution.

In the diffractogram of the washed product with HCl (Figure 5), the samples C07H and C10H had a difference in the 2θ peaks representing SiC. The diffractogram of sample C07H showed peaks for the 3C-SiC polytype while C10H showed peaks for the 3C-SiC and 2H-SiC polytypes. Gao et al. (2011) reported the formation of the 2H-SiC polytype prepared with the MR method [12]. The formation of 2H-SiC is influenced by the porosity of the carbon base material. In solids chemistry, carbonization can change the porosity and crystallinity of carbon. Furthermore, Solihudin et al. (2015) stated that the carbonization of rice husk char at 900 °C caused graphitization [13]. This also applies to rice husk char carbonized at 1000 °C. The regularity of the structure in graphite is presumably one of the factors for the formation of 2H-SiC. Aside from graphitization, the base material also affects the formation of the 2H-SiC polytype. The gasified rice husk char has a carbon morphology shielded by SiO₂; hence, the template for the MR process is carbon. This causes a pseudomorphic transformation with template C, and the SiC crystal structure is estimated to follow the C crystal structure.

In addition, the diffractogram result from the washing with HCl also indicated the presence of diffraction peaks probably from Mg₂SiO₄ and Silicon. Sadique (2010) stated that Mg₂SiO₄ could not be washed with HCl except at the washing stage with a mixture of 5% HF: 4.38 M CH₃COOH (1:9).

3.3. Calcination of Reduction and Purification Products with Mixed Acid Solutions

The reduction product after washing with a 1 M HCl solution was further calcined in the next stage. Calcination was carried out in atmospheric air at a temperature of 700 °C to remove residual carbon. The calcined product showed a color change from black to gray, which indicated that the remaining carbon had been burned. FTIR results of the calcined products in the air atmosphere indicated the presence of SiO₂ (Figure 6 and Table 4).

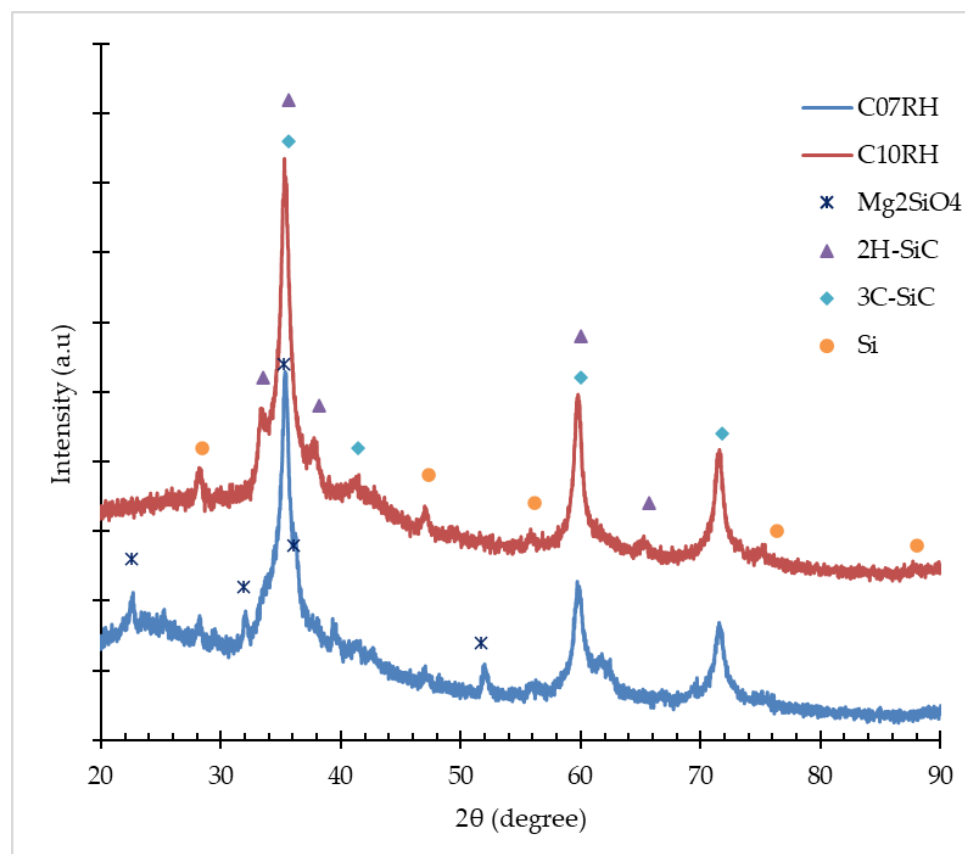


Figure 5. Diffraction pattern of product from washing with 1 M HCl.

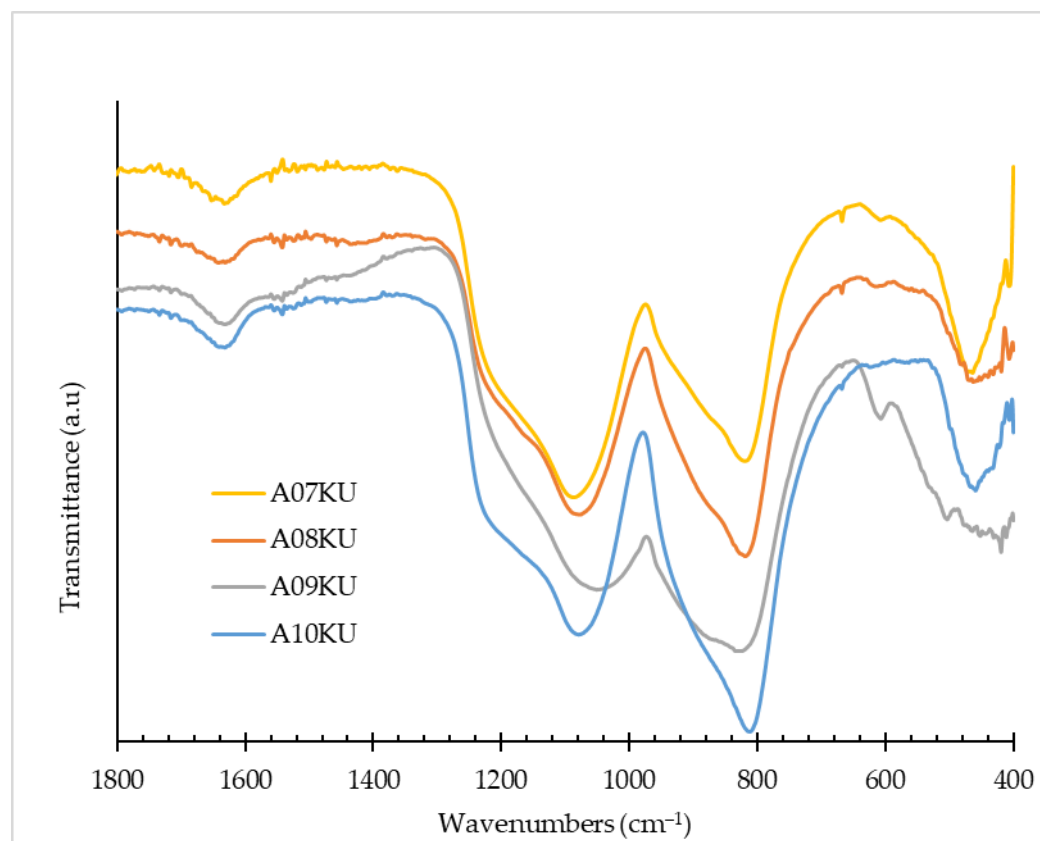


Figure 6. IR spectrum of the reduction product after calcination in air atmosphere at 700 °C.

Table 4. Comparison of the absorption of the reduction products after calcination in an atmosphere of air at a temperature of 700 °C.

Type of Vibration	Wavenumber/cm ⁻¹				
	C07KU	C08KU	C09KU	C10KU	Standard
Bending vibration of ≡Si-O at (≡Si-O-Si≡)	464	462	-	459	465–475 [14]
Stretching vibration of Si-C	820	819	834	812	830 [17]
Asymmetric stretching vibration of =Si-O at (=Si-O-Si=)	1087	1077	1051	1079	1050–1150 [14]

According to the characterization results of the calcined product with XRD (Figure 7), the shape of the diffractogram was relatively the same as that of the washing product with HCl. This indicates that the calcination process at 700 °C did not change the crystal structure and the crystalline components contained in the sample are the same. Furthermore, the composition analysis results on the diffractogram in Table 5 show that the SiC products after calcination decreased compared to after washing with HCl. This indicates that some of the SiC turned into SiO₂ during the calcination process in the presence of air. This is in line with the results of characterization by FTIR, which shows the presence of SiO₂.

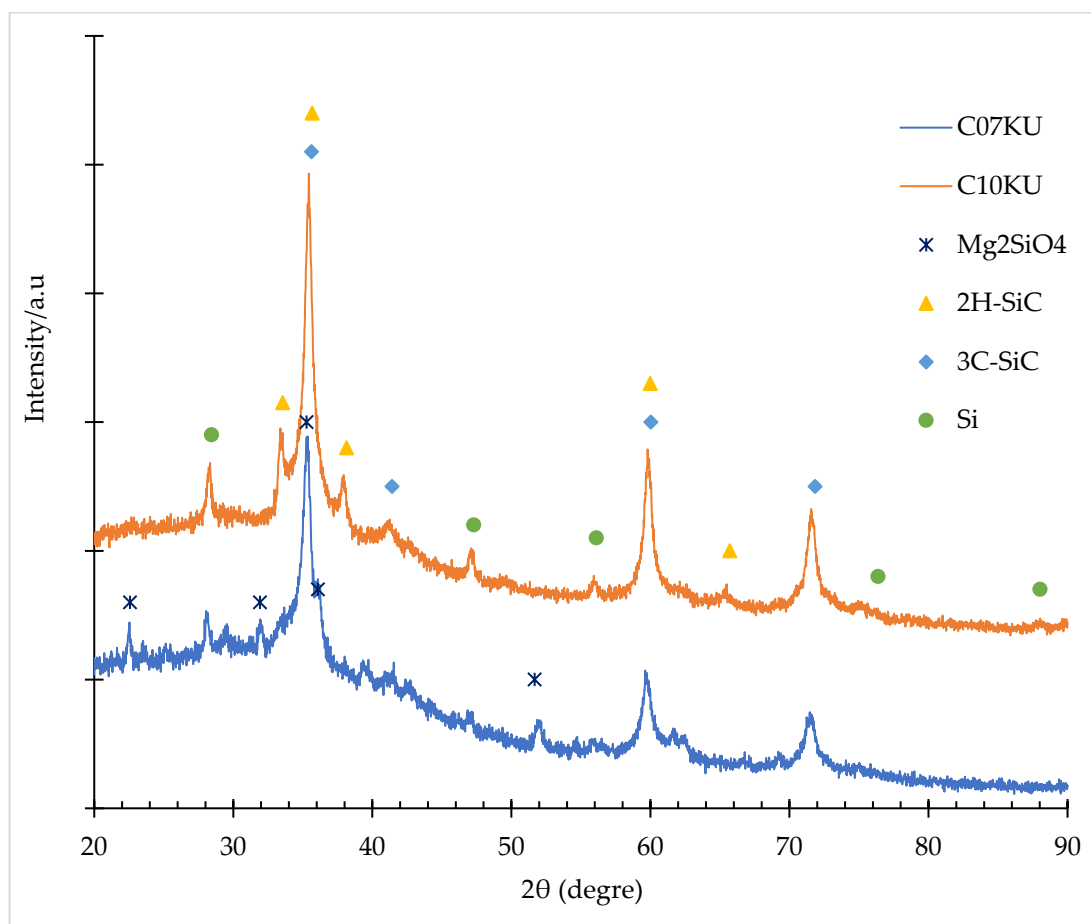


Figure 7. Diffraction pattern of calcined products in air atmosphere at 700 °C.

Washing with a mixture of 5% HF: CH₃COOH 4.38 M (1:9) served to remove silica compounds; hence, the SiC obtained was free from impurities. Figure 8 and Table 6 show that in all the IR spectra, an absorption band was observed at a wave number of 830 cm⁻¹, which is a Si-C stretching vibration, and there was no absorption band from the silica compounds. This shows that SiO₂ and Mg₂SiO₄ in the calcined product can be washed with a mixture of 5% HF: CH₃COOH 4.38 M. The absence of Mg₂SiO₄ was further

demonstrated in the XRD characterization results as indicated by the loss of the diffraction peak at 2θ around 22.6° , 32.0° , 36.1° , and 51.7° in all diffractograms (Figure 9). Although silicon could not be removed at this stage, there was still a diffraction peak at 2θ around 28.4° , 47.2° , and 56.1° in all samples.

Table 5. Comparison of compound composition of HCl washing product and air calcined product.

Compound	Composition/% (w)			
	Before Calcination		After Calcination	
	C07RH	C10RH	C07KU	C10KU
3C-SiC	32.1	43.6	27.3	42.5
2H-SiC	-	17	-	18.1
Si	2.7	4	3.4	3.3
Mg ₂ SiO ₄	26.6	-	26	-
Total Amorphous	38.6	35.5	43.3	36.2

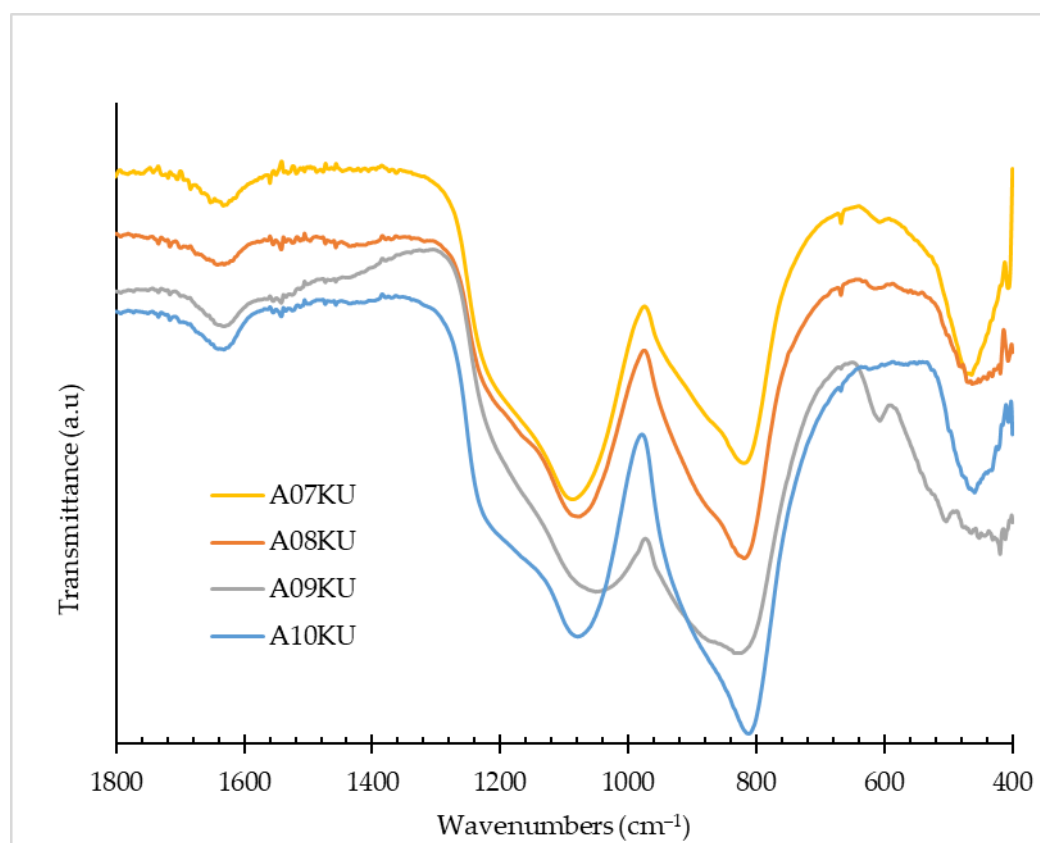


Figure 8. IR spectrum of the calcined product in an air atmosphere at 700 °C after washing with a mixture of HF and CH₃COOH solutions.

Table 6. IR absorption of calcined products at a temperature of 700 °C in air atmosphere after washing with a mixture of HF and CH₃COOH solutions.

Type of Vibration	Wavenumber/cm ⁻¹				
	C07SiC	C08SiC	C09SiC	C10SiC	Standard
Stretching vibration of Si-C	822	822	829	818	830 [17]

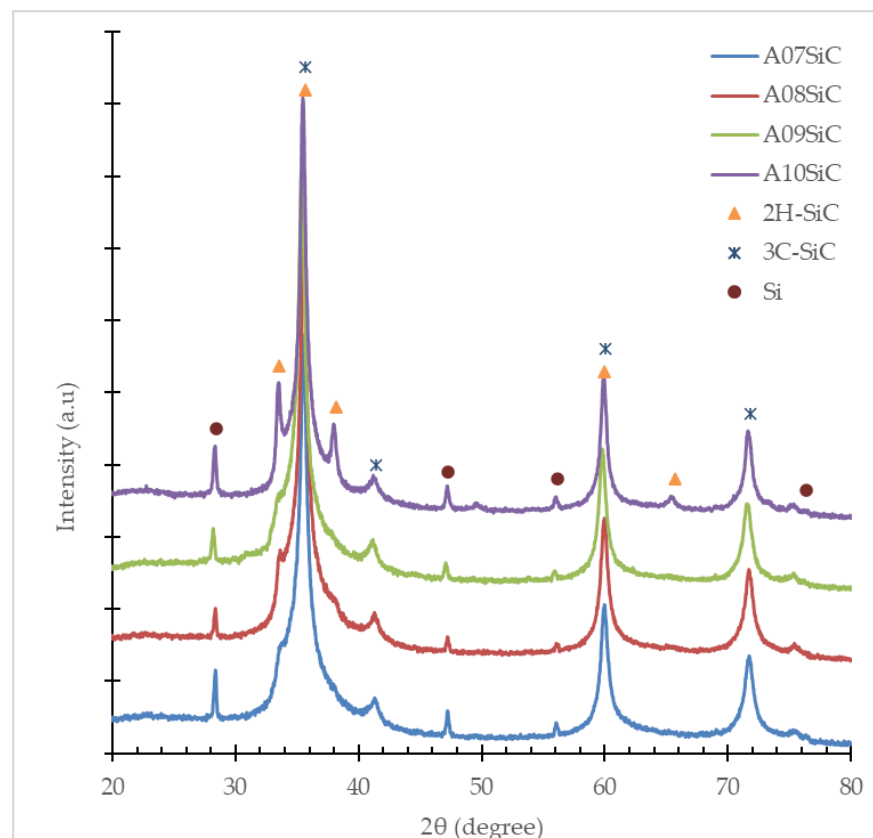


Figure 9. Diffraction pattern of the calcined product at 700 °C in an air atmosphere after washing with a mixture of HF and CH₃COOH solutions (based on rice husk char from carbonization at 700 to 1000 °C).

The search results using the “High Score Plus” software on the diffractograms as presented in Figure 9 show that the SiC formed is a mixture of 3C-SiC and 2H-SiC polytypes with a dominant composition of 3C-SiC. The composition of the 2H-SiC polytype formed from carbonized rice husk charcoal at 700 °C, 800 °C, and 900 °C was almost the same. Meanwhile, SiC from the rice husk char carbonized at 1000 °C was a mixture of 3C-SiC and 2H-SiC with the highest composition of 2H-SiC compared to the other synthesis products. The composition of each component is listed in detail in Table 7.

Table 7. Composition of the calcined product at 700 °C in an air atmosphere after washing with a mixture of HF and CH₃COOH solutions.

Sample	Composition/wt.%		
	3C-SiC	2H-SiC	Si
C07SiC	87.9	9.7	2.4
C08SiC	88.9	9.8	1.3
C09SiC	89.5	9.7	0.8
C10SiC	82.3	15.5	2.2

Figure 10 is a photograph of the SEM characterization of the calcined product after washing from C07SiC and C10SiC samples. Based on the SEM photogram, the calcined products showed various particle sizes. The size of the SiC particles formed appeared to depend on the particle size of the rice husk charcoal used. The photogram also shows that the particle structure resembles the structure of rice husk char. The elemental composition in the selected area according to the results of EDS (Figure 11 and Table 8) confirmed that apart from the presence of Si and C elements, Mg and O were also present as impurities.

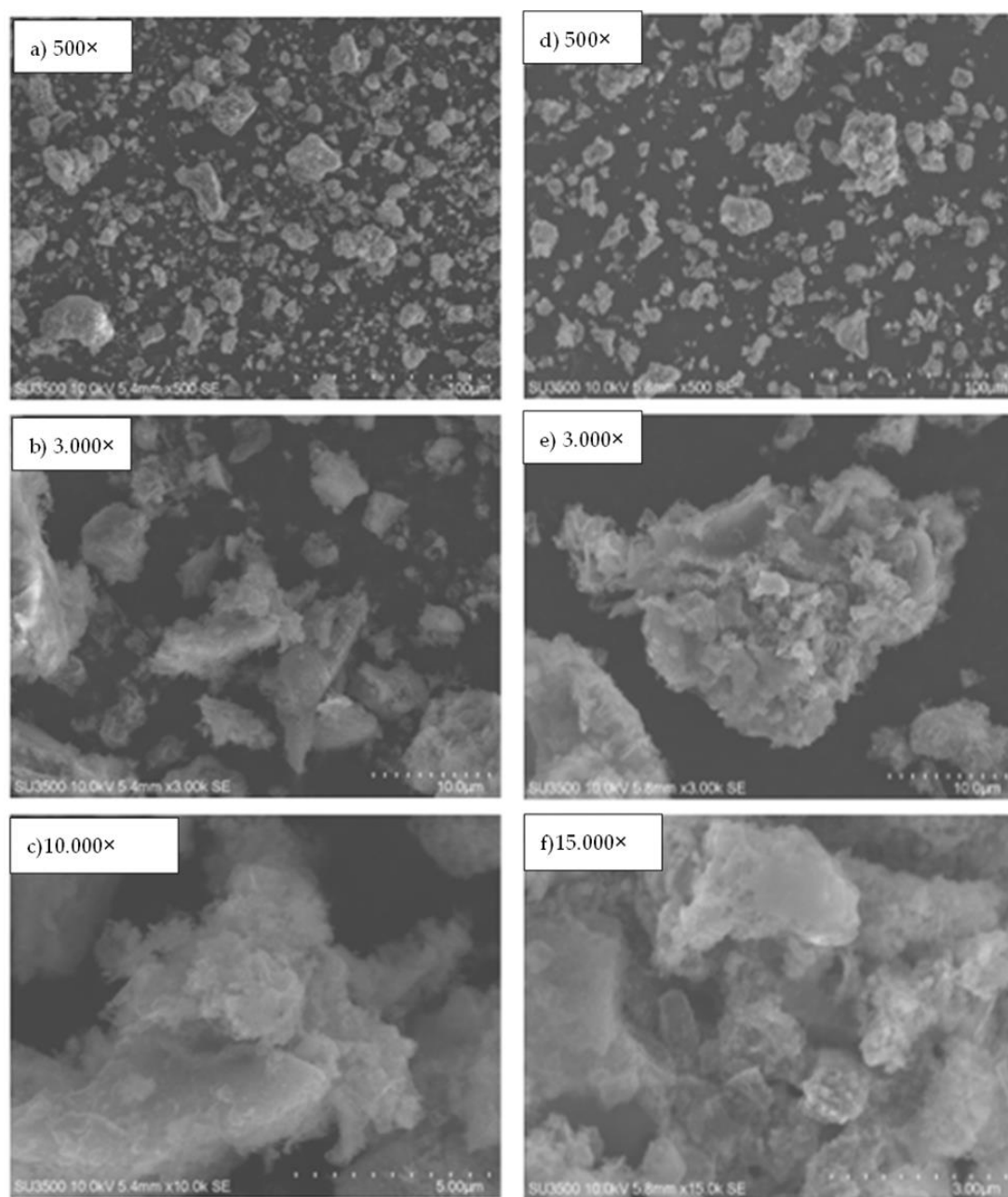


Figure 10. SEM photographs: (a–c) C07SiC, (d–f) C10SiC.

Table 8. Elemental composition of C07SiC and C10SiC (wt.%).

Element	C07SiC		C10SiC	
	Area 1	Area 2	Area 1	Area 2
C	64.26	66.82	73.78	71.44
O	12.53	15.76	10.98	7.12
Mg	0.74	1.49	0.37	0.49
Si	22.46	15.93	14.86	20.96

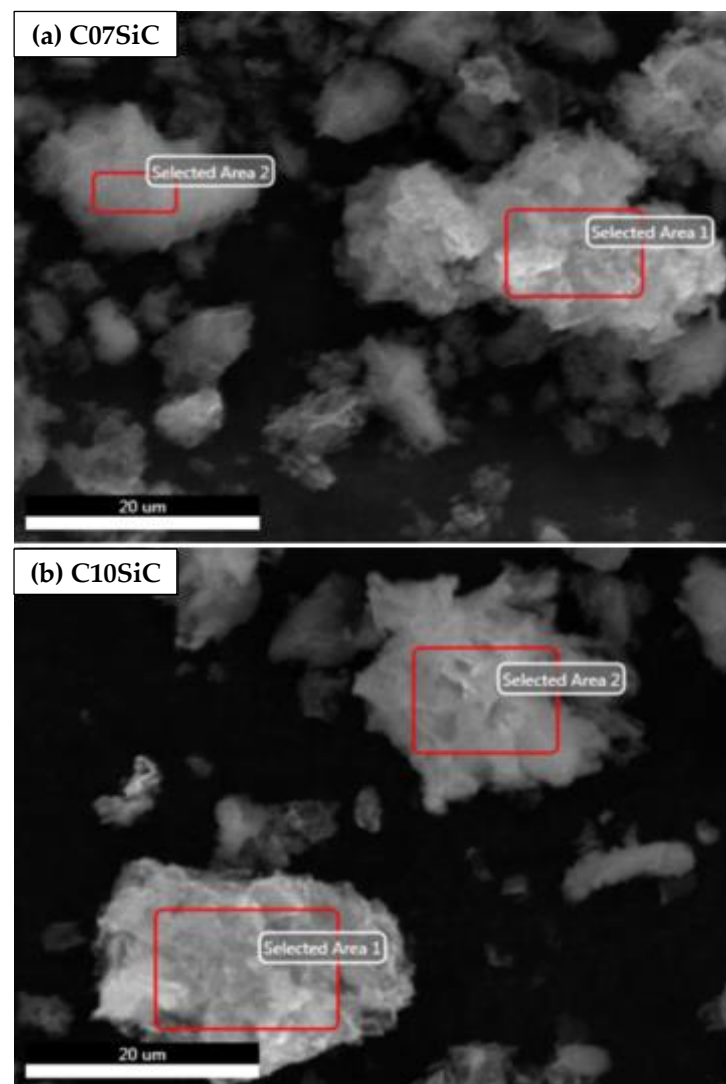


Figure 11. EDS photogram: (a) C07SiC, (b) C10SiC.

4. Discussion

The carbonization of rice husk charcoal has succeeded in increasing the degree of carbonization of rice husk charcoal. The increase in the degree of carbonization is indicated by the decreasing intensity of IR absorption at wave numbers $1400\text{--}1500\text{ cm}^{-1}$, which is associated with aromatic rings, and 1600 cm^{-1} , which is associated with ketone, aldehyde, lactone, and carboxyl groups. Silica was found in all the rice husk charcoal samples (before and after carbonization). This was indicated by the presence of a strong band located from the IR absorption around 803 and 1088 cm^{-1} .

SiC was formed using the magnesiothermic reduction method on all rice husk charcoal samples from all variations of carbonization temperatures. However, there are still impurities as a result of side reactions in the form of MgO, Mg_2SiO_4 , and Si. The washing step with an HCl solution for the reduction results succeeded in separating the MgO impurities. In addition, the washing step with the HCl solution also affected the SiC polytype formed. From the magnesiothermic reduction of rice husk, the 3C-SiC polytype was produced, whereas SiC with polytypes 3C-SiC and 2H-SiC was produced from the reduction of carbonized rice husk charcoal at $1000\text{ }^\circ\text{C}$. Impurities in the form of Mg_2SiO_4 and Si cannot be separated at this washing step with an HCl solution.

The calcination stage in air at $700\text{ }^\circ\text{C}$ for the reduction product after washing with the HCl solution succeeded at reducing the residual carbon, as indicated by the color change in the calcination product from black to gray. Follow-up action in the form of washing

with a mixture of acids (HF and CH_3COOH) succeeded at separating SiO_2 and Mg_2SiO_4 impurities, but Si was not able to be separated. In addition, at this stage, a mixture of 3C-SiC and 2H-SiC polytypes was produced from rice husk charcoal at all variations of SiC carbonization temperatures, where 3C-SiC was more dominant.

This work delivered a new insight on how the SiC formation can be carried out with a more feasible and versatile method. However, some improvements to eliminate impurities must still be conducted in the future, yet the method already informed us about the effect of the carbonization temperature on the formation of SiC, which can be beneficial for future work. Therefore, in the future, the ultimately pure SiC can be obtained through an optimized carbonization temperature.

5. Conclusions

The use of carbonized rice husk char at a temperature of 1000 °C as the base material for the formation of SiC using the magnesiothermic reduction method produced 2H-SiC polytypes; hence, it can be adopted as a new synthesis route. In the SiC formation process using the magnesiothermic reduction method based on rice husk char, the by-products included MgO, Mg_2SiO_4 , and Si. MgO and Mg_2SiO_4 were successfully separated by acid washing, while Si was not separated from the SiC product.

Author Contributions: Conceptualization, Writing—Original Draft Preparation, Writing-Review and Editing Supervision, Investigation, Formal Analysis, Resources, Data Curation, Funding Acquisition, S.S.; Supervision, Formal Analysis, Writing—Review and Editing, Writing-Original Draft, H.H.; Writing—Review and Editing, Supervision, Formal Analysis, A.R.N.; Writing—Review and Editing, A.A.U. All authors have read and agreed to the published version of the manuscript.

Funding: This study was supported by The National Research and Innovation Agency, Kemenristek/BRIN, through the PDUPT Grant with contract no. 1207/UN6.3.1/PT.00/2021.

Data Availability Statement: Data will be available upon request for specific reason.

Acknowledgments: The authors thanks to Yoga Trianzar Malik for his assistance formatting the manuscript.

Conflicts of Interest: The authors declared no conflict of interest.

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