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Sol-gel synthesis of boron carbide from a polyvinyl borate precursor: effect of heat treatment temperature on phase formation

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ABSTRACT

In this study, boron carbide (B₄C) was synthesized via the sol-gel method using polyvinyl alcohol (PVA) and boric acid (H₃BO₃) as precursor materials. After complete dissolution of PVA in water, boric acid was added to the solution, and the resulting mixture was filtered and subjected to pyrolysis at 600 °C. The pyrolyzed precursor was subsequently heat-treated at 1200 °C, 1300 °C, and 1400 °C under an argon atmosphere. The phase evolution and structural characteristics of the synthesized products were analyzed by Fourier transform infrared spectroscopy (FT-IR) and X-ray diffraction (XRD). Thermal behavior was evaluated using thermogravimetric analysis (TGA), while the microstructural features of selected samples were examined by scanning electron microscopy (SEM). The XRD results indicated that boron carbide formation becomes more pronounced with increasing heat-treatment temperature. In particular, the sample treated at 1300 °C exhibited diffraction peaks corresponding to rhombohedral B₄C. TGA results showed relatively lower mass loss for this sample, suggesting improved thermal stability compared with the other samples. In contrast, the sample treated at 1400 °C exhibited changes in mass loss behavior that may be associated with the formation of additional phases during thermal analysis under a nitrogen atmosphere. Overall, the results suggest that heat treatment at 1300 °C provides favorable conditions for the formation of boron carbide within the investigated temperature range. The sol-gel route employed in this study offers a simple and potentially energy-efficient approach for the synthesis of boron carbide powders.

I. INTRODUCTION

Advanced ceramic materials are high-performance engineering materials that exhibit exceptional wear resistance in applications requiring high mechanical properties at elevated temperatures. Boron carbide (B₄C) is one of the most prominent examples of advanced ceramics due to its high melting temperature (approximately 2400 °C), low density (2.52 g/cm³), high hardness, high elastic modulus (approximately 448 GPa), excellent resistance to chemical corrosion, and high neutron absorption cross-section. Owing to these outstanding properties, boron carbide has become one of the most extensively studied materials for advanced technological applications [1-4]. As a result, it is widely preferred in aerospace, space, defense, medical, and microelectronic applications [5-6].

There are several different routes for the synthesis of boron carbide (B₄C), including carbothermal reduction, magnesiothermal reduction, synthesis from elemental precursors, vapor-phase reactions, and polymer-derived precursor methods [7-9].

Currently, commercial production of boron carbide mainly relies on mechanical alloying, thermal regeneration of activated carbon, and carbothermal reduction processes. However, each of these techniques has inherent disadvantages. These limitations include difficulties in controlling particle size and morphology, a high risk of contamination, and challenges in obtaining a high-purity final product [10-11]. For instance, the carbothermal reduction method involves the reduction of boric acid with carbon at high temperatures in electric furnaces.

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Nevertheless, this process often results in a significant amount of residual free carbon in the final product.

Moreover, achieving fine particle sizes requires costly and challenging milling processes, and the method necessitates high-temperature furnace operations. Since temperatures above 2000 °C are required, precise temperature control using electronic systems becomes extremely difficult. In addition, non-uniform heating in electric arc furnaces may lead to incomplete reactions, resulting in impurity-related issues in the final product [12-13].

In another study, Mirabelli et al.[14] reported the synthesis of boron carbide using polyvinyl pentaborane as a precursor. In their work, polyvinyl pentaborane oligomers were obtained by thermal polymerization of 2-(H₂C = CH)B₅H₈. Subsequently, these oligomers were converted into boron carbide by heat treatment at 1000 °C for 8 h under an argon atmosphere. The boron carbide obtained in this study exhibited high ceramic strength; however, the high cost of the raw materials makes this method commercially unattractive.

Although these synthesis methods have been successful at the laboratory scale, the development of a synthesis route suitable for large-scale production of boron carbide structures remains necessary. In this context, the sol–gel method has emerged as one of the most extensively investigated techniques for the production of high-quality ceramic materials[15,16]. This method involves the formation of a homogeneous solution (sol) through hydrolysis and condensation reactions of metal alkoxides or metal salts, which are referred to as molecular precursors[17,18]. With the continuation of the reaction, polymerization leads to the formation of a three-dimensional network structure and gelation. The desired material density is achieved by applying heat treatment to remove solvents and undesired compounds from the system [19-21].

Boron carbide obtained via this synthesis route exhibits high ceramic strength; however, the high cost of raw materials limits its commercial feasibility. In addition, boranes are highly reactive and potentially hazardous starting materials. Therefore, researchers have increasingly focused on readily available, non-hazardous, and low-cost materials. The primary role of materials used together with boric acid is to supply the carbon atoms required for boron carbide synthesis. In this regard, polyols have attracted considerable attention. B–O–C borate esters obtained using different polyols show great promise to produce B₄C powders. For esterification purposes, glycerin [22], citric acid [23], cellulose [24], glucose [9], and polyvinyl alcohol (PVA) [25] have been widely used.

However, despite the extensive use of different polyols, many of these systems still require relatively high processing temperatures or result in incomplete phase formation. In contrast, PVA-based precursor systems provide a more homogeneous carbon distribution and improved control over the carbon-to-boron ratio, which can significantly influence the phase evolution during heat treatment.

There is significant interest in studies where PVA is employed as a carbon source for the production of boron carbide [26]. Accordingly, the sol–gel synthesis of boron carbide (B₄C) using PVA and boric acid (H₃BO₃) has emerged as a promising alternative production route. In this approach, polyvinyl borate (PVBO) is first formed through a condensation reaction between PVA and boric acid. During subsequent pyrolysis, PVA is carbonized while boric acid is converted into boron oxide, leading to the formation of carbon and boron oxide species that later participate in the carbothermal formation of boron carbide.

In this study, boron carbide (B₄C) powders were synthesized via the sol–gel method using a polyvinyl alcohol (PVA)–boric acid precursor system. The obtained polyvinyl borate (PVBO) precursor was first pyrolyzed at 600°C

and subsequently heat-treated at different temperatures (1200, 1300, and 1400°C) under an argon atmosphere. The aim of this study is to investigate the effect of heat treatment temperature on the phase formation, thermal stability, and microstructural evolution of boron carbide synthesized from this precursor system. The obtained products were systematically characterized using FT-IR, XRD, TGA, and SEM analyses in order to determine the optimum conditions for boron carbide formation.

II. EXPERIMENTAL METHOD

2.1 Materials

Boric acid (99.5% purity), supplied by Eti Maden, was used as the boron source. Polyvinyl alcohol (PVA, Mowiol® 4-88, $M_w \approx 31,000$), obtained from Aldrich, was employed as the carbon source. Pyrolysis and subsequent heat treatments were carried out in high-alumina crucibles under an argon atmosphere.

2.2 Synthesis of Polyvinyl Borate (PVBO)

Polyvinyl borate (PVBO) was synthesized through the dehydration and condensation of the product formed by the reaction between PVA and boric acid. For this purpose, 100 g of PVA was added to 1000 mL of distilled water at 80 °C and stirred for 30 min until complete dissolution was achieved. In a separate beaker, 80 g of boric acid (H_3BO_3) was dissolved in 500 mL of distilled water at 80 °C. These two solutions were then combined and stirred for 1 h, resulting in the formation of a white PVBO gel. The obtained PVBO gel was dried in a crystallizing dish at 200 °C for approximately 2 h until the water within the gel was completely evaporated. Subsequently, the dried product was ground to obtain PVBO powder.

Polyvinyl borate (PVBO) is formed through the reaction of B–OH groups originating from boric acid with the –OH groups of PVA. In this structure, B–O–C bonds are formed within the PVA hydroxyl side chains, which remain water-soluble [27]. This reaction enables the incorporation of boron atoms into the organic polymer matrix. The PVBO synthesis reaction and the idealized structure of PVBO are illustrated in Figure 1.

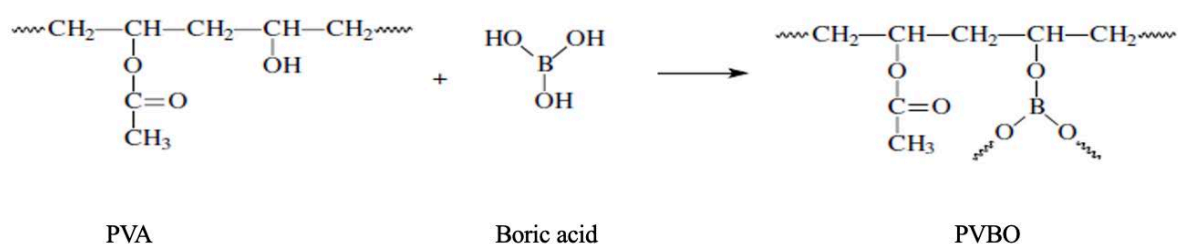


Figure 1. Condensation Reaction Of PVA And Boric Acid Leading To The Formation Of PVBO [28]

2.3. Pyrolysis of Polyvinyl Borate Samples

This process was carried out to enrich PVA in carbon and to remove water originating from boric acid. PVBO powders were placed in high-alumina crucibles and pre-pyrolyzed at 200 °C for 2 h. In the second stage, the main

pyrolysis treatment was performed, which also helps to minimize residual carbon in the final product. The PVBO powders contained in high-alumina crucibles were pyrolyzed at 600 °C for 2 h. This process was conducted under an argon atmosphere with a gas flow rate of 4 LPM and a heating rate of 10 °C/min.

2.4. Heat Treatment of the Pyrolyzed Samples

The pyrolyzed PVBO samples placed in high-alumina crucibles were subjected to heat treatment at three different temperatures. The first sample was heat-treated at 1200 °C, the second at 1300 °C, and the third at 1400 °C. All heat treatments were carried out under an argon atmosphere with a gas flow rate of 4 LPM and a heating rate of 10 °C/min. The samples were held at the selected temperatures for 5 h.

The exact role of the pyrolysis step in facilitating carbothermal reduction is not yet fully understood; however, it is widely accepted that pyrolysis promotes the formation of reactive carbon and boron oxide species, which are essential for boron carbide formation. Pyrolysis is defined as the thermochemical decomposition of organic materials in the absence of oxygen at high temperatures [29]. During the pyrolysis process, boric acid is converted into boron oxide, while PVA undergoes carbonization, leading to the formation of boron oxide and carbon. These products subsequently participate in the synthesis of boron carbide. One of the advantages of pyrolysis is the removal of undesirable elements and residual carbon from the compound. Consequently, boron carbide crystals can be produced at relatively lower temperatures with a minimal amount of free carbon [30].

After the pyrolysis of PVBO, boron carbide is synthesized via carbothermal reduction through the reaction between carbon and B₂O₃ [31]. The carbothermal reduction reaction is given in Equation (1).



According to the carbothermal reduction reaction given in Eq. (1), the formation of boron carbide requires a specific carbon-to-boron oxide ratio. The stoichiometric C/B₂O₃ molar ratio for B₄C formation is approximately 3.5. However, in practical synthesis routes, slightly higher carbon contents are generally required to compensate for carbon losses during pyrolysis and to ensure complete reduction of boron oxide.

In the present study, polyvinyl alcohol (PVA) was used as the carbon source, while boric acid (H₃BO₃) served as the boron precursor. Based on thermogravimetric considerations and previous studies, the initial composition was selected as 100 g PVA and 80 g H₃BO₃, corresponding to a weight ratio of 5:4. This composition was chosen in order to obtain a suitable carbon-to-boron oxide ratio after pyrolysis at 600 °C, which is favorable for the carbothermal synthesis of boron carbide with minimal residual free carbon.

Based on the selected precursor composition, the stoichiometric relationship between boric acid and the carbon source was evaluated. A total of 80 g of H₃BO₃ corresponds to approximately 1.294 mol of boric acid. During dehydration, boric acid converts into boron oxide according to the reaction:



Thus, this amount of H₃BO₃ theoretically produces 0.647 mol of B₂O₃, corresponding to approximately 45.0 g of B₂O₃. According to the carbothermal reduction reaction given in Eq. (1), the formation of boron carbide requires

a C/B₂O₃ molar ratio of 3.5. Therefore, the complete reduction of this amount of B₂O₃ requires approximately 2.265 mol of carbon, corresponding to about 27.2 g of carbon.

Considering the repeating unit of polyvinyl alcohol (C₂H₄O)_n, the theoretical carbon content of PVA is approximately 54.5 wt%. Accordingly, 100 g of PVA can theoretically provide up to 54.5 g of carbon if fully carbonized. This amount exceeds the carbon required for the carbothermal reduction of boron oxide derived from 80 g of boric acid. Therefore, the selected PVA/H₃BO₃ ratio (5:4) provides a sufficient carbon source for boron carbide formation, although the actual carbon yield after pyrolysis may be lower due to the release of volatile decomposition products. The overall experimental procedure is schematically illustrated in Figure 2.

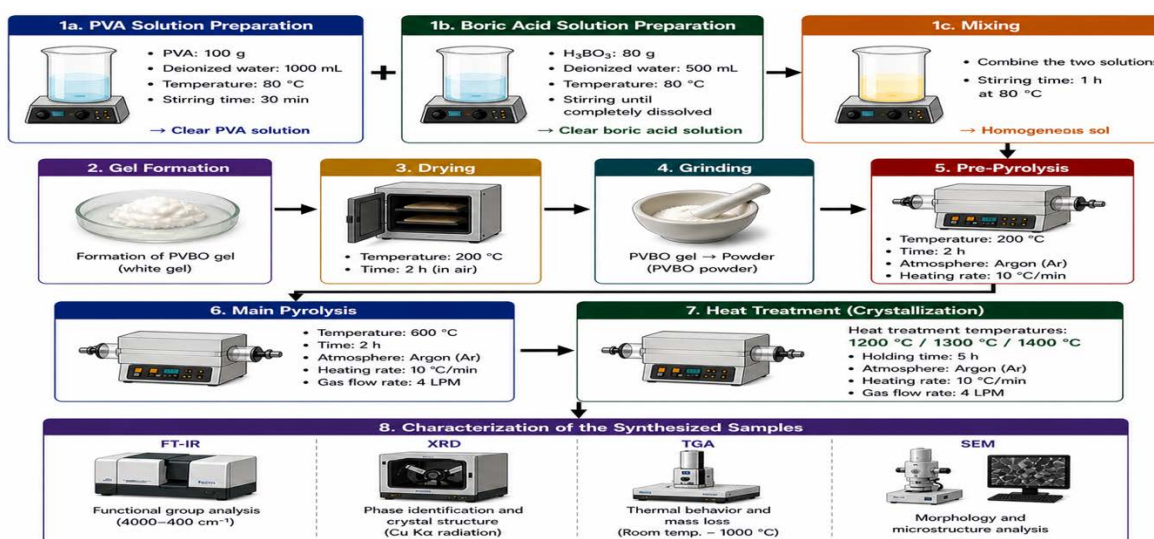


Figure 2. Schematic representation of the sol-gel synthesis route employed for the preparation of boron carbide (B₄C) powders using polyvinyl alcohol (PVA) and boric acid (H₃BO₃) precursors

III. RESULTS AND DISCUSSIONS

3.1. Fourier Transform Infrared (FT-IR) Analysis

The FT-IR spectra of PVA, boric acid, PVBO precursor, and the heat-treated samples are presented in Figure 3. As shown in Figure 3b, the spectrum of PVA exhibits a broad absorption band at approximately 3247 cm⁻¹, which corresponds to the O–H stretching vibration of hydroxyl groups. In the PVBO spectrum, this band shifts to around 3201 cm⁻¹ and its intensity decreases, indicating the interaction between the hydroxyl groups of PVA and boric acid during the formation of the polyvinyl borate structure.

In addition, characteristic absorption bands related to boron-containing structures appear in the PVBO spectrum. The band observed at approximately 1198 cm⁻¹ is attributed to B–O–H deformation vibrations, while the band at around 1021 cm⁻¹ corresponds to the B–O–C stretching vibration, indicating the formation of borate ester bonds in the PVBO structure.

As shown in Figure 3a, the FT-IR spectra of the heat-treated samples reveal several characteristic absorption bands. The absorption band located around 1424 cm⁻¹ is associated with B–O stretching vibrations. Furthermore, the band observed near 937 cm⁻¹ can be attributed to B–O–C stretching vibrations associated with borate structures,

suggesting the presence of boron–oxygen–carbon linkages in the intermediate structures formed prior to the carbothermal formation of boron carbide [32].

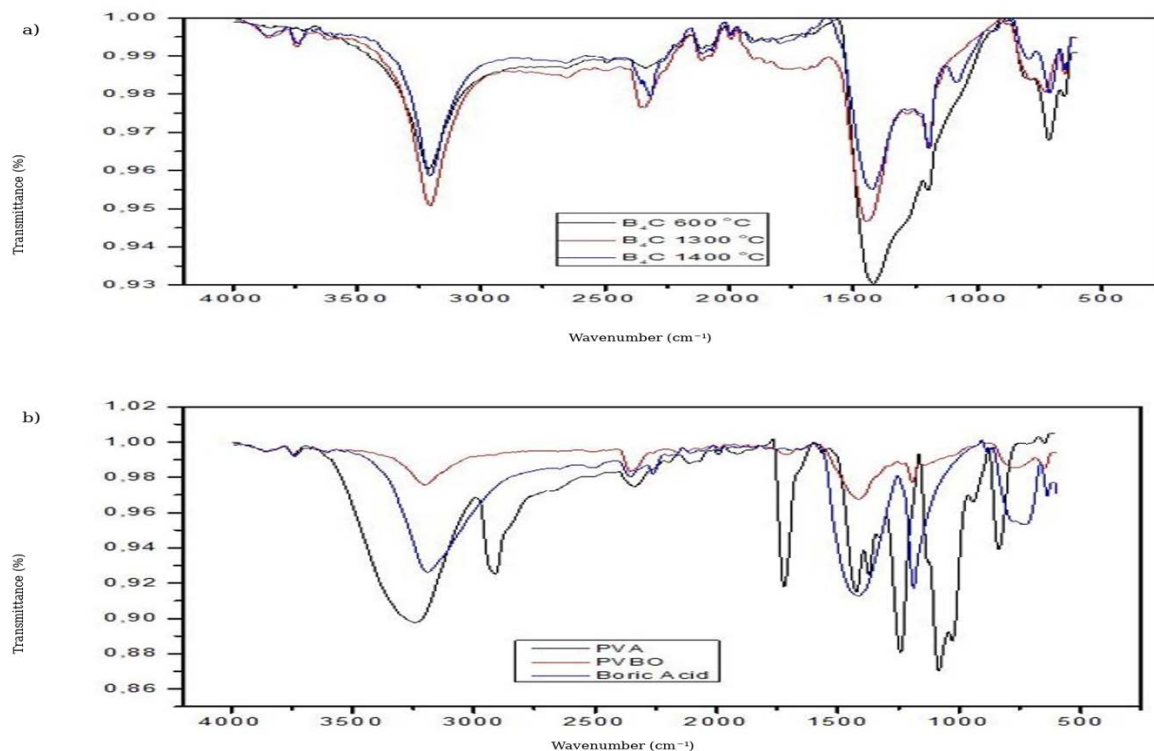


Figure 3. FT-IR spectra of the samples: **(a)** heat-treated samples at 600, 1300, and 1400 °C, and **(b)** precursor materials including PVA, PVBO, and boric acid

3.2. Thermogravimetric Analysis (TGA)

The thermogravimetric analysis (TGA) curves of the pyrolyzed sample and the heat-treated samples are presented in Figure 4. The measurements were carried out under a nitrogen atmosphere with a heating rate of 10 °C min⁻¹.

As shown in Figure 4, the sample pyrolyzed at 600 °C without further heat treatment exhibits a significant mass loss during the heating process. The total mass loss reaches approximately 45% up to 1400 °C, with a considerable portion of this loss occurring at relatively low temperatures around 200 °C. This behavior can be attributed to the removal of residual volatile components and the decomposition of intermediate structures formed during the sol-gel process.

For the heat-treated samples, improved thermal stability is observed compared with the pyrolyzed sample. The specimen heat-treated at 1200 °C shows a higher mass loss than the sample treated at 1300 °C, indicating that the latter exhibits relatively greater thermal stability.

A notable feature of the thermograms is observed for the sample treated at 1400 °C, which shows a mass loss of approximately 20% around 200 °C, nearly twice that observed for the sample treated at 1300 °C. The mass continues to decrease up to about 800 °C, followed by a slight mass increase that persists until approximately 1180 °C. This behavior may be related to reactions occurring between boron-containing species and nitrogen during the analysis, potentially leading to the formation of boron nitride at elevated temperatures.

Overall, the TGA results suggest that the sample heat-treated at 1300 °C exhibits relatively improved thermal stability compared with the other samples.

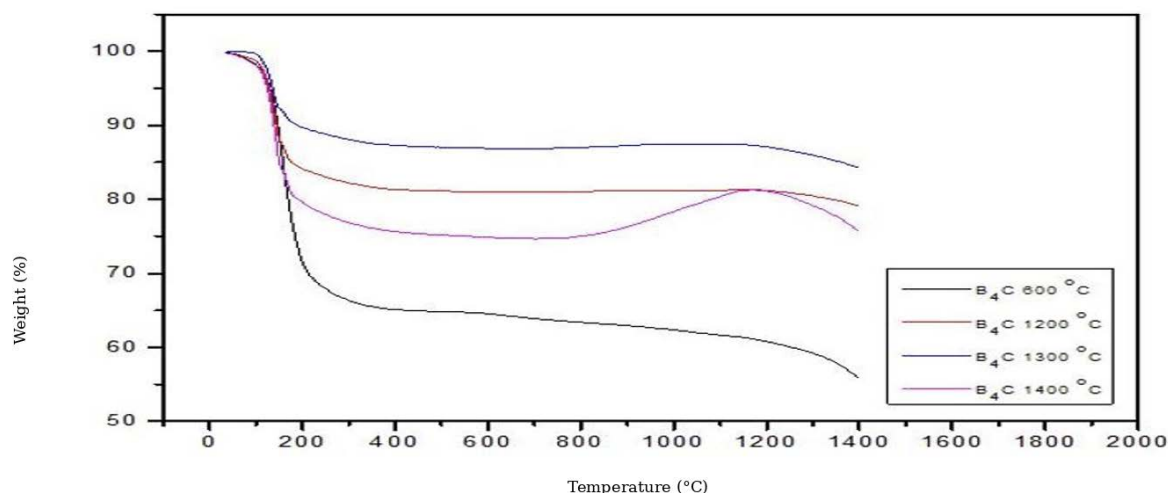


Figure 4. TGA thermograms of the samples heat-treated at different temperatures

3.3. XRD Analysis of Polyvinyl Borate (PVBO)

The X-ray diffraction (XRD) patterns of the synthesized samples are presented in Figure 5. The diffraction patterns were recorded using a Rigaku Ultima IV diffractometer in the 2θ range of 5–90°. The PVBO precursor (Figure 5a) exhibits broad diffraction features, indicating the predominantly amorphous nature of the precursor structure formed by the condensation reaction between PVA and boric acid.

After pyrolysis at 600 °C (Figure 5b), the diffraction pattern remains largely amorphous, suggesting that the polymeric precursor decomposes to form a mixture of carbon and boron oxide rather than a well-crystallized boron carbide phase. This observation is consistent with the formation of an intermediate structure during the pyrolysis stage.

In contrast, the sample heat-treated at 1300 °C (Figure 5c) shows the emergence of diffraction peaks corresponding to the characteristic reflections of rhombohedral boron carbide (B_4C). The main diffraction peaks located at approximately 23°, 35°, 38°, and 60° are consistent with the standard diffraction data reported in the ICDD database for B_4C . The appearance of these peaks indicates that the carbothermal reaction between carbon and boron oxide leads to the formation of boron carbide at this temperature.

For the sample treated at 1400 °C (Figure 5d), the diffraction pattern exhibits sharper and more intense reflections, indicating an increase in crystallinity. However, additional diffraction peaks are also observed, suggesting the formation of boron-rich carbide phases or related boron–carbon phases at elevated temperature. Therefore, although higher temperature enhances crystallization, it may also promote further phase evolution beyond the preferential formation of the B_4C phase.

Under the present experimental conditions, the XRD results indicate that heat treatment at 1300 °C provides more favorable conditions for the formation of the target B_4C phase.

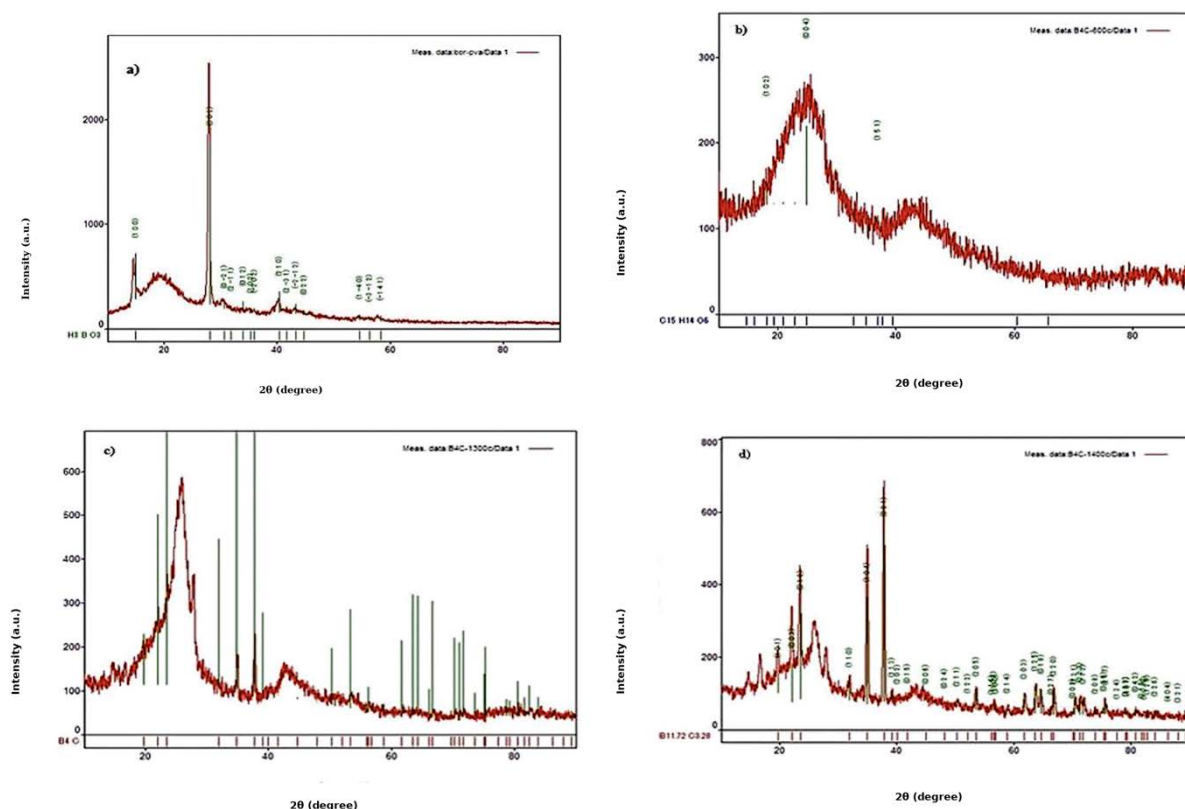


Figure 5. XRD patterns of the synthesized samples: (a) PVBO precursor, (b) PVBO pyrolyzed at 600 °C, (c) sample heat-treated at 1300 °C, and (d) sample heat-treated at 1400 °C

3.4. Morphological Investigation

The morphological characteristics of the samples obtained after the pyrolysis process and subsequent heat treatment were examined by scanning electron microscopy (SEM), and the corresponding images are presented in Figure 6. The SEM observations were performed for the sample pyrolyzed at 600 °C and for the sample heat-treated at 1400 °C for 5 h under an argon atmosphere.

As shown in Figure 6a, the sample pyrolyzed at 600 °C exhibits a sponge-like porous structure formed as a result of the decomposition of organic components in the sol-gel system during the pyrolysis stage [33]. The presence of spherical particles within the porous matrix has been reported in the literature to be associated with boric acid (H_3BO_3) residues in polymer-derived precursor systems. Previous studies have shown that these boric acid particles can be removed through washing with hot water[34].

After heat treatment at 1400 °C for 5 h under an argon atmosphere (Figure 6b), the microstructure changes noticeably. The spherical particles observed after pyrolysis are no longer visible, and the structure appears more porous and agglomerated. This morphological evolution indicates the decomposition of residual precursor phases and structural rearrangement during the high-temperature treatment, which is consistent with the carbothermal formation of boron carbide.

Although SEM observations were performed for the pyrolyzed sample and the sample treated at 1400 °C, the formation of boron carbide at 1300 °C was primarily confirmed through XRD analysis, where the characteristic diffraction peaks of rhombohedral B_4C were observed.

Despite the advantages of the sol-gel method, certain limitations should be considered. These include relatively long processing times, multiple heat treatment steps, and challenges in controlling porosity and residual carbon content. Therefore, further optimization of the process parameters is required for large-scale applications.

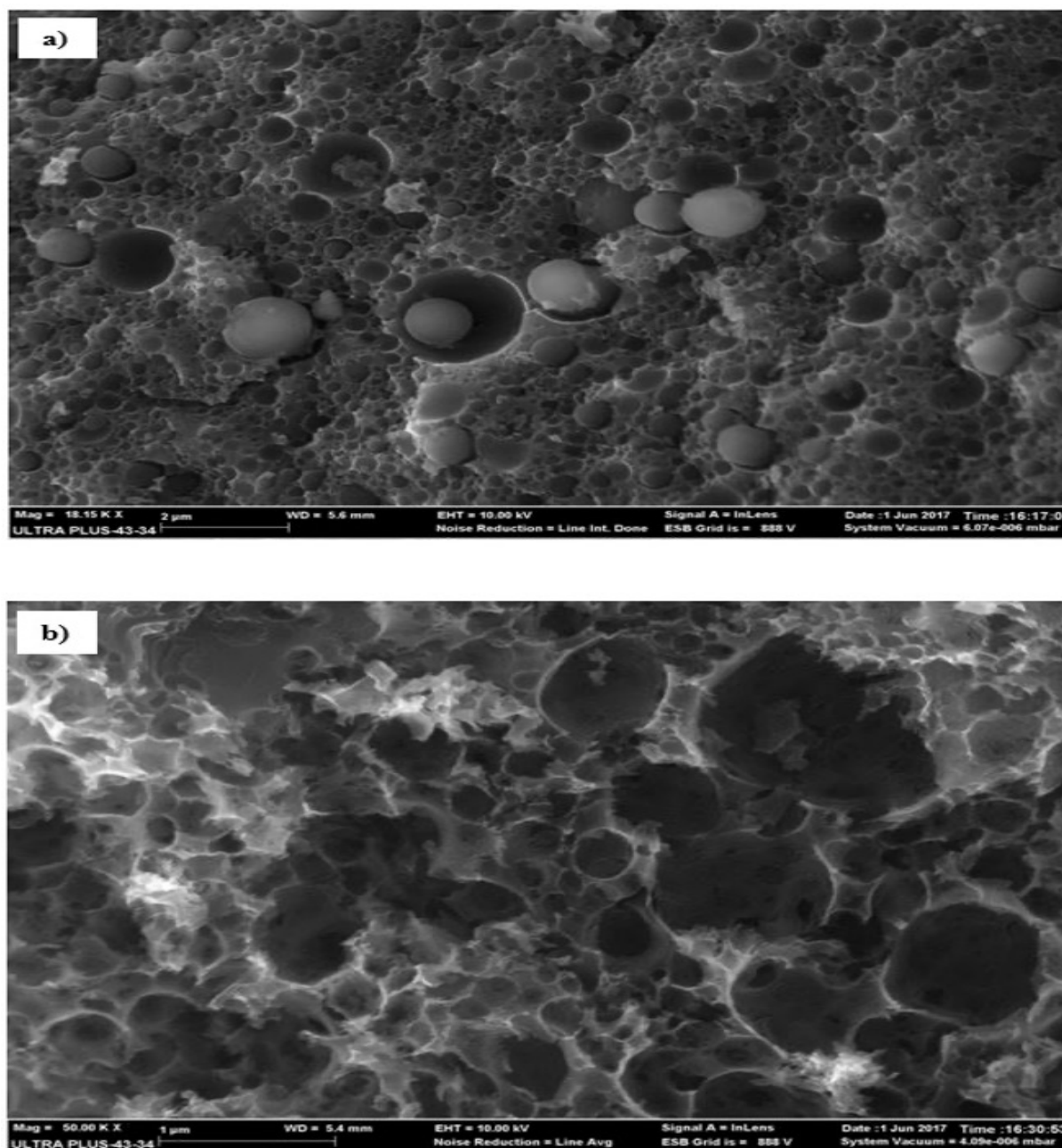


Figure 6. SEM images of the synthesized samples: (a) sample pyrolyzed at 600 °C for 2 h, showing a sponge-like porous structure; (b) sample heat-treated at 1400 °C for 5 h under an argon atmosphere

IV. CONCLUSIONS

In this study, boron carbide (B_4C) was synthesized via the sol-gel method using polyvinyl alcohol (PVA) and boric acid (H_3BO_3) as precursor materials. The obtained gel structures were first pyrolyzed at 600 °C and subsequently heat-treated at 1200 °C, 1300 °C, and 1400 °C under an argon atmosphere. The influence of heat-

treatment temperature on phase formation and thermal behavior was systematically investigated using FT-IR, XRD, and TGA analyses.

The XRD results revealed that the formation of boron carbide becomes more pronounced with increasing heat-treatment temperature. In particular, the sample treated at 1300 °C exhibited diffraction peaks corresponding to rhombohedral B₄C, indicating successful formation of boron carbide through the carbothermal reaction. The FT-IR analysis also supported the evolution of boron-containing bonding structures at elevated temperatures.

Thermal analysis showed that the sample treated at 1300 °C exhibited relatively lower mass loss compared with the other samples, suggesting improved thermal stability. It should be noted that the TGA measurements were conducted under a nitrogen atmosphere. For the sample treated at 1400 °C, the mass change behavior suggested the possible formation of additional phases due to interactions between boron-containing species and nitrogen at elevated temperatures.

Overall, the results indicate that heat treatment at 1300 °C provides favorable conditions for the formation of boron carbide within the investigated temperature range. The sol-gel route employed in this study represents a simple and potentially energy-efficient approach for producing boron carbide powders with relatively homogeneous precursor distribution.

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