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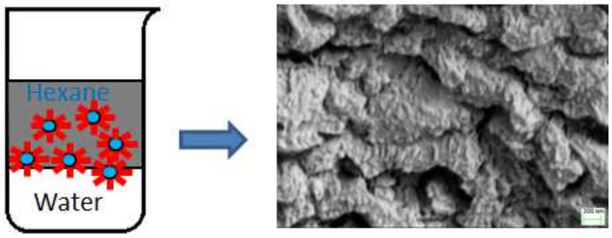

Nano boric acid crystal fabrication under combination effect of hexane and dispersant.

Improvement of Crystallization and Particle Size Distribution of Boric Acid in the Processing of a Boron Carbide Precursor

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Abstract

In the processing of crystalline boron carbide, boron oxide and carbon precursors are used as feedstock into a carbothermal reduction process reactor. The uniformity of the precursor blends has a dramatic effect on the homogeneity of resultant boron carbide. In boron carbide precursor preparation via a wet process, boric acid is usually dissolved into water at 80 °C and then mixed with carbon and dried at 120°C, followed by calcination then comminution to obtain the desired feed particle size. However, controlling the crystallization and particle size of boric acid is challenging and there are no prior published studies dealing with this issue. In this study, a simple process demonstrated that adding hexane to the boric acid water solution during the drying can achieve a highly crystallized powder with a narrow particle size distribution. Hexane generates the supersaturation during the recrystallization of boric acid by increasing boric acid nucleation. The hexane can inhibit boric acid crystal growth by isolating the formed nuclei thus providing a barrier to molecular diffusion. The water to hexane volumetric ratio, suitable processing parameters, and optimized dispersant amount were investigated. It was found that the process of mixing the optimum solution at room temperature and then drying it at 120°C produced the relatively uniform 0.3 μm particles. By adding 1.5 wt% dispersant (Triton X-100) to the mixture, 20 nm boric acid particle size can be obtained. The boron carbide powder fabricated by carbothermal process from the modified procursor demonstrated a finer and more uniform particle size with lower free carbon content. The precursor calcination temperature could be lowered from 600°C to 450°C.

Key Words: boric acid, crystallization, particle size, boron carbide precursor

Introduction

Boron carbide is a superhard ceramic that is used for diverse applications due to special properties such as high melting point (2720 K), high hardness (32-35 GPa Knoop hardness), low density (2.52 g cm^{-3}), high Young's modulus (450–470 GPa), and shear modulus (180 MPa) [1-3]. The combination of these properties makes it beneficial for applications ranging from abrasive powders and wear resistance parts to ceramic armor [4-5]. Boron carbide powders are typically produced by carbothermal reduction of a precursor in which boric acid and carbon are mixed and then heated to high temperatures. The synthesized boron carbide powder properties highly depend on the particle size and homogeneity of the precursor powder. The precursor prepared with well-dispersed boric acid and carbon black improves the reaction between the two phases and lowers the reaction temperature. This results in a finer and more uniform boron carbide powder, which renders lower sintering temperatures and better mechanical properties [6-7]. The general boron carbide precursor process requires making the premixed powder first, where carbon black and boric acid [4, 8-9] are mixed in water at 80°C, at which the boric acid's solubility is much higher than at room temperature, followed by calcination, crushing and sieving to get the desired particle size. The carbon black particle size in premixed powder is relatively easy to control. There are already many studies on dispersing carbon black in an aqueous solution by adding surfactant [10-12]. However, controlling the particle size of boric acid has not yet been realized, since boric acid can recrystallize during drying and continually grow into large particles.

In ceramic fabrication, particle size control strategies can be found in review literature [13-22]. Principally, these methods take advantage of chemical routes that can be mainly classified into coprecipitation, sol-gel, hydrothermal reaction, emulsion and pyrolysis. Coprecipitation is the process that precipitates several metal cations by adding a precipitation agent to reduce the source aqueous solution with controlled pH. The sol-gel method uses a chemical solution (sol) or colloidal particles to produce an integrated network (gel). The typical chemical source for the sol-gel process is metal alkoxides. The formation of metal oxide involves the generation of metal-oxo or metal-hydryoxo polymer in solution. Hydrothermal reactions are carried out in autoclaves that heat the binary solution to about 180 – 230°C. Microemulsions are the mixed solution of oil, water and surfactant. The aqueous phase may contain salt while the oil phase can be a complex of different hydrocarbons and olefins. Gas phase synthesis usually involves the pyrolysis of the metal gas source or reaction with O_2. However, the above-mentioned particle size control methods are difficult to apply to boric acid. The low solubility of boric acid in water eliminates the coprecipitation and emulsion methods. The sol-gel synthesis is not feasible because of the lack of a boron alkoxide source. Hydrothermal reactions and gas phase pyrolysis need expensive equipment to complete the reaction, which limits mass fabrication. From our knowledge, there is no report on the synthesis of narrowly distributed boric acid particles.

The drying process for a boric acid and carbon mixture is actually a dehydration course where crystallite formation of boric acid is under supersaturation by evaporative crystallization [23]. The control of crystallite size in evaporative crystallization is usually achieved by precise regulation of the evaporation rate [24-25]. This method is mostly employed in pharmaceutical fabrication or very special materials and is usually not applied to large scale ceramic raw materials mixing processes. The particle size control of boric acid in carbide precursors has not been studied.

Recently, Miller et. al. showed a rapid carbothermal reduction process which produced boron carbide that was chemically pure with fine grain size by directly mixing carbon black and boric acid as a precursor [26]. It is believed that the homogeneity of the precursor can be further improved to meliorate the produced boron carbide powder. This study examines the control of crystal growth by introducing an immiscible second liquid phase into the boric acid and water system that the boric acid does not dissolve and therefore acts as a crystal growth inhibitor during crystallization. This phase can cover the crystallite surface to minimize the contact between crystallite and water when the crystallization begins to occur, lower the boric acid solubility, and inhibit crystallite growth in water. Alkanes are considered good candidates because alkanes are inexpensive chemicals, they evaporate easily during drying, they are immiscible with water, and at the same time they do not dissolve boric acid. This work presents a simple process to improve the particle size distribution of the resultant mixture while maintaining the high boric acid crystallization by using hexane in the boric acid-water system. The crystallization of boric acid in a water-hexane system is similar to a water-oil system where the hexane (like an oil phase) can inhibit the crystal growth by partly covering the particle surface as the boric acid begins to crystallize. The synthesized boron carbide could validate the contribution from this modified precursor.

Results and Discussion

The Effect of the Water-to-Hexane Ratio

Fig. 1 is the XRD pattern of dried boric acid powder from the mixture of different volumetric ratios of water to hexane as described in the experimental section. During the drying, the boric acid (H_3BO_3) and metaboric acid (HBO_2) crystals are formed as the water evaporates[27].

Among the H_3BO_3 peaks, the 28.1° diffraction has the strongest intensity. The XRD pattern shows the relationship between hexane concentration and the degree of boric acid crystallization. Generally, the narrower and higher peak indicates a higher degree of crystallization, which can be reflected in XRD peak intensity counts and full width at half maximum (FWHM). The dehydrated sample without hexane (i.e., the regular drying procedure) demonstrates high crystallization but with a crystal size (about 0.6 μm) as shown in Fig. 2 (a). The morphology of irregular and imperfect hexagonal crystals indicates the anisotropic crystal growth. With the addition of a small amount of hexane (samples S1, S2, and S3), the degree of crystallization was relative low (low intensity in XRD pattern). As more hexane was added (samples S4 to S8), the degree of crystallization increased and the highest crystallized sample was S6 (i.e., 50 ml water to 60 ml hexane). The peaks' counts and FWHM at $2\theta=28.1°$ shown in Table 1 also confirmed this conclusion. Though there are no data in literature on the relationship between boric acid crystallization degree and synthesized boron carbide particles, highly crystallized boron trioxide may form from high crystallized boric acid, which can easily transfer to boron carbide crystal through reconstruction (a more energy saving route) during boron carbide synthesis.

Fig. 2 shows the SEM images of samples including that without hexane (sample S0) and those particles with the addition of hexane (samples S3 to S9). Table 2 is the record of the particle size distribution from line profile based on SEM images. As discussed before, the sample without hexane presents particle size range (0.1 μm to 0.6 μm) and a wide distribution (in 0.1-0.2, 0.2-0.4, 04-0.6 μm, respectively) accompanied by non-perfect crystallized morphology. This confirms the nonuniform crystal growth in the regular precursor process. By adding hexane (S1 to S3), SEM images (S1 and S2 images not showed here) demonstrate major amorphous phases with small amount irregular shape particles. For the comparison, SEM of S3 is presented. These precipitated irregular shape particles demonstrate low degree of crystallization within the range of 0.1-0.6 μm with unimodal distribution. As the hexane ratio increases, well-crystallized hexagonal structures are observed. From sample S4 to S6, the fully crystallized hexagonal structure becomes more dominant. In samples S4 and S5, the particle size varies from 0.1 μm to 0.6 μm with multimodal distribution in three ranges (0.1-0.2, 0.2-0.4, 04-0.6 μm, respectively), while sample S6 has the unimodal distribution in the more narrow range (0.2-0.4 μm). Hexane contributes to particle size reduction and narrow distribution by surrounding the precipitated boric acid crystal during drying, which leads to oversaturation and inhibits the crystal growth. As the hexane ratio further goes up (samples S7 and S8), particles distribute become wide. For S7, particles still in the range 0.1-0.6 μm but numbers in three ranges are close. In S8, particles grow into large crystal with wide distribution. Too less or too much hexane results in large particle size distributions. In these two cases, local supersaturation is higher than the suitable ratio of hexane resulting in more fluctuation in the mixture [30].

The Effect of the Mixing Procedure

To find the best mixing procedure, three mixing processes were carried out: M1, M2 and M3. Based on the results shown in above section, the volumetric ratio of water to hexane was set the same as in sample S6 (50 ml : 60ml).

SEM images of samples from different mixing procedures are shown in Fig. 3. The particle size distribution analysis is also showed in Table 3. The M1 procedure produced highly crystallized particles with a narrow particle distribution in 0.2 to 0.4 μm, while the M2 and M3 procedures produce the multimodal particle size distribution in three ranges (0.1-0.2, 0.2-0.4, and 0.4-0.6 μm). In the M1 procedure, the precipitated boric acid crystallite was under lower local supersaturation fluctuation during dehydration [28]. Temperature plays an important role in this kind of system [29-30]. When lowering the hexane solution to 70 °C, the minor temperature difference between two solutions decreases the supersaturation degree so the droplet size can grow relatively easy. As for dropping to room temperature, the flocculating constituent is formed when a drop falls into hexane solution, so hexane has relatively little effect on the solubility during drying [30].

The Effect of Dispersant

As discussioned above, the optimal water-to-hexane volumetric ratio and the suitable mixing procedure have been explored. Though highly crystallized fine particles were achieved, the agglomerated particles, which counteract the benefits of the small particle size of boric acid, were still present in all samples. Therefore, the effect of dispersant amount will be examined in order to find the optimal dispersion effect. Triton X-100 is a nonionic surfactant that can be utilized in many different systems without changing the solution pH value to reach the best dispersed condition. Different amounts of dispersant were added to the above optimized procedure in order to compare dispersion effect.

As shown in Fig. 4, the 0.5 wt% dispersant was obviously not enough to separate the stacked particles (Fig. 4(a)). The particle size varied from 150 nm to 400 nm. As the dispersant increased to 1.0 wt%, the bulk began to separate into agglomerated parts (Fig. 4(b)). The observed average particle size was about 200 nm and the particle distribution was more uniform that that of 0.5 wt% dispersant. When the dispersant was further increased to 1.5 wt%, a particle of 20 nm with highly-uniform distribution was observed (Fig. 4(c)). Further addition of the dispersant generates particle agglomeration with non-uniform particle size distribution because of the repulsion between dispersant molecules (Fig. 4(d) and (e)). The particle distribution of boric acid with dispersant only, i.e., no hexane added, also displayed the relatively narrow particle size distribution at 1.5 wt% Triton X-100 but average particle size is about 200 nm (Fig. is not shown). However, the crystallization degree without the hexane is fairly low (Fig. 5). The corresponding peak intensity and FWHM at 28.1° are 15927, 0.173 for the sample with hexane and 6157, 0.189 for the sample without hexane, respectively. This result indicates that the existence of hexane helps boric acid crystallization.

Comparison of Fabricated Boron Carbide Powders

The boron carbide powders were fabricated from both the typical precursor and nano boric acid-based precursor by carbothermal reduction. The XRD pattern from Fig. 6 reveals that the free carbon amount (marked with arrow, other peaks all from boron carbide) reduces from 1.3 wt% to 0.36 wt%. Furthermore, the calcination of nano boric-acid based precursor can be conducted at 450°C compared to 600°C for regular precursor. SEM images from several different magnifications confirm that synthesized boron carbide powder is finer (1 μm) and more uniform when using nano boric acid-based precursor rather than regular precursor. The typical SEM image is present in Figure 7 for comparison.

The decrease of free carbon and the narrowing of particle size distribution in the synthesized boron carbide is attributed to nano size effect and precursor homogeneity, since the reacted powder had the same chemical composition as shown in Table 4 (regular precursor 29.81 wt% carbon and nano precursor 29.84 wt% carbon). While an effective method to quantify the precursor homogeneity, carbon contents in the sieved fine and coarse powder may also provide a measurement of precursor uniformity. As seen in Table 2, the nano precursor has a smaller carbon content difference (2.6%) than the regular precursor (5.3%). The well-dispersed nano-size boric acid and carbon black could improve the two-phase contact and facilitate the transformation to boron carbide, and therefore lower the free carbon amount.

Experimental

A simple route has been developed by using hexane as the second liquid phase without complicating the general boron carbide precursor fabrication. The experiment included three parts: studying the water-to-hexane volumetric ratio on boric acid crystallization and the effects of different mixing procedures, and identifying the best dispersant concentration. In the general procedure (denoted M1), we dissolved 5g boric acid powder (Borax Inc.) into 50 ml distilled deionized water (corresponding to 1.62 molar/L), then added different volumes of hexane (Fisher). The mixed solution was heated to 80°C to dissolve the boric acid powder until a clear solution was observed, then it was dried at 120°C. The samples' nomenclature is shown in Table1. In the second mixing procedures (denoted M2), two solutions were prepared: one was a 1.62 molar/L boric acid water solution at 80°C, and another one was 1.2 times water volume of hexane solution set at 70°C. The boric acid solution was then added to hexane solution with a dropwise titration (one droplet is about 0.3ml, the rate of dropwise is 24ml/min) and dried at 120 °C. The third procedure (denoted M3) was identical with M2 except that the hexane solution was set at room temperature.

To further improve the particle distribution, the effect of surfactant was examined. Triton X-100, a nonionic surfactant, was used since nonionic surfactants can be applied to different systems without changing the solution pH as ionic surfactants do. The selected amounts were 0.5 wt%, 1 wt%, 1.5 wt%, 2.0 wt% and 2.5 wt% of boric acid weight. The crystallization was evaluated by XRD (PANalytical, Model PW3040/60) with Cu Kα radiation (λ = 1.5406 Å) at a scanning rate of 0.1 degree/min. The full width at half maximum (FWHM) in XRD pattern is only evaluated to the peak at 28.1°, which is associated with the boric acid (H_3BO_3) phase. Particle morphology was observed by a scanning electron microscope (SEM, Zeiss Gemini 982). The particle size distribution was analyzed by manually measuring the particle size in different directions from series of SEM images and then tabulated. For making boron carbide powder, the precursor composition consisted of 7 molar carbon black with 6 molar boric acid (50% excess boric acid). In typical precursor formula, 3.0 wt% Triton X-100 (based on carbon black weight) was added. For the modified precursor (this experiment), the dispersant amount of carbon black was kept at 3.0 wt%, while additional dispersant was selected and added to the optimal experimental result of boric acid. The premixed powder was then calcinated in a tube furnace. The typical calcination schedule was set at 600°C for 2 hours while calcinations of the modified powder mixture were conducted at 450°C for 2 hours. After calcinations, the product was crushed and sieved (coarse powder, 125-425 μm) to maintain powder flowability. The fine powder (less than 125 μm) was removed. The total carbon content of sieved fine and coarse powders was measured by hot extraction method (LECO CS230 Carbon and Sulfur Analyzer). The sieved powders were fed into a rapid carbothermal furnace (Thermal Technologies Inc., Santa Rosa, CA, USA) by screw feeder with the hot zone temperature set at 1850 °C in a similar process described by Miller et al. [26].

Conclusion

A simple process for controlling boric acid crystallization and particle size in boron carbide precursor preparation has been developed by adding an amount of hexane to the initial suspension. The mixing procedure and the dispersant amount was optimized in this study. It was found that the optimal procedures for premixed powders entailed mixing 1.62 M boric acid water solution with 1.2 times the water volume hexane solution, dissolving the boric acid at 80°C, and then drying it at 120°C. The results demonstrate that well-crystallized boric acid with uniform particle distribution can be fabricated with average particle size of about 20 nm when 1.5 wt% dispersant (Triton X-100) is added into the above solution. The modified process greatly enhances the boric acid crystal formation and particle size distribution and further contributes to a lower calcination temperature. The synthesized boron carbide powder from the modified precursor has a better particle size distribution and lower free carbon content compared to boron carbide powders prepared for normal precursor.

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Table Captions

Table 1: Solvent volume for the samples used in the premixed powder and corresponding XRD peak parameters

Table 2. Measured particle size distribution through line profile the SEM image for different water-to-hexane ratios

Table 3. Measured particle size distribution through line profile the SEM image for different mixing precedures

Table 4: Comparison of carbon contents (wt%) for coarse and fine precursor materials

Figure Captions

Figure 1. The XRD pattern of boric acid with different volumetric ratio of water-to-hexane

Figure 2. The particle size and morphology of dried boric acid powder with different volumetric ratios of water to hexane (typical grains are highlighted with borders): (a) S0, (b) S3, (c) S4, (d) S5, (e) S6, (f) S7, and (g) S8

Figure 3. SEM images of powders made from three different mixing procedures with a fixed water to hexane ratio at 50 ml: 60 ml: (a) M1, (b) M2, and (c) M3

Figure 4. Particle size distribution of the premixed powder with different amounts of dispersant: (a) 0.5 wt%, (b) 1 wt%, (c) 1.5 wt%, (d) 2.0 wt%, and (e) 2.5 wt%

Figure 5. XRD pattern of boric acid powder with 1.5 wt% dispersant with and without hexane

Figure 6. Comparison of XRD pattern of boron carbide powder from regular precursor (bottom) and nano boric acid based precursor (top)

Figure 7. SEM images of B4C powder synthesized from the regular precursor (left) and nano boric acid-based precursor (right)

Table 1

Sample	Water volume (ml)	Hexane volume (ml)	Counts (at 2θ=28.1°)	FWHM (at 2θ=28.1°)
S0	50	0	17602	0.167
S1	50	10	1119	0.322
S2	50	20	3233	0.265
S3	50	30	2542	0.219
S4	50	40	11998	0.198
S5	50	50	14811	0.187
S6	50	60	15446	0.181
S7	50	70	12153	0.184
S8	50	80	14952	0.196

Table 2

Samples	Particle Counts in different size rang (μm)				
	0.1 – 0.2 (μm)	0.2 - 0.4 (μm)	0.4- 0.6 (μm)	0.6- 0.8 (μm)	0.8- 1.0 (μm)
S0	40	65	33	1	
S3 *	23	67	18	4	
S4	61	89	23	2	
S5	31	49	23	4	1
S6	16	135	4		
S7	39	60	30	7	
S8 **	< 0.5 (μm)	0.5 – 1.5 (μm)	1.5 – 2.5 (μm)	2.5 – 3.5 (μm)	> 3.5 (μm)
	2	4	2	6	1

*: Sample S3 only can be observed about 10 vol% particles without clear crystal structure

**: Particles size ranges are different with other samples

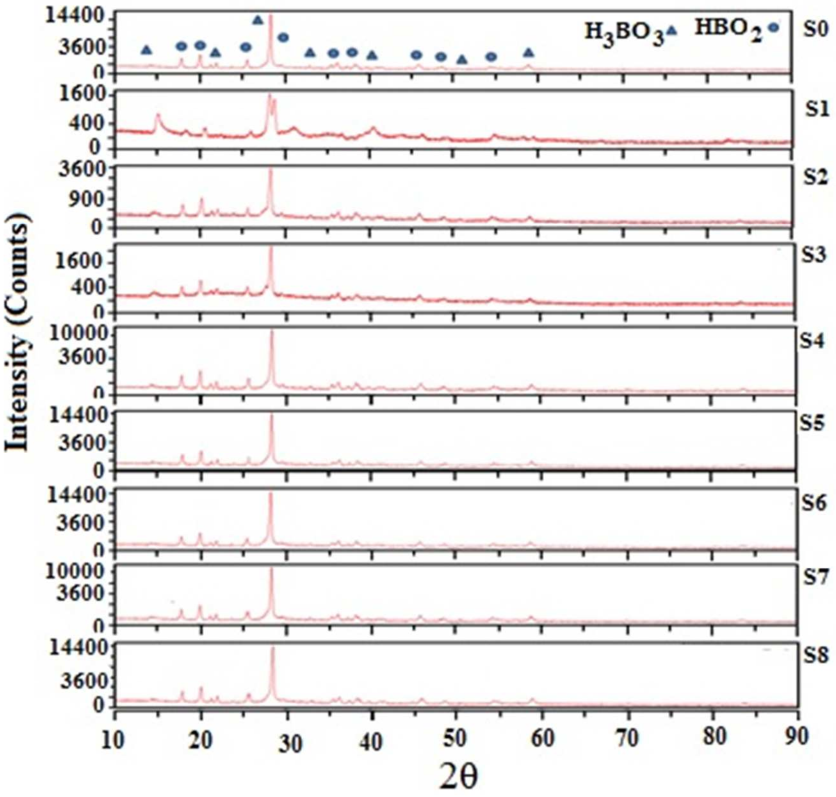
Table 3

Samples	Counts in different size rang (µm)				
	0.1 – 0.2 (µm)	0.2 - 0.4 (µm)	0.4- 0.6 (µm)	0.6- 0.8 (µm)	> 0.8 (µm)
M1 *	16	135	4		
M2	86	49	19	4	21
M3	135	92	7	5	6

* M1 is the same as sample S6

Table 4

Total Carbon and Calculation	Samples			
	CL with (50% more) H_3BO_3 calcinated at 600°C regular		CL with (50% more) nano H_3BO_3 calcinated at 450°C	
	Fine	Coarse	Fine	Coarse
Carbon Contents in precursor (wt%)	28.32	29.81	29.08	29.84
Relative Difference (%) of Carbon Contents in Precursor	5.3		2.6	
Free Carbon (wt%) in Produced Boron Carbide	1.3		0.36	



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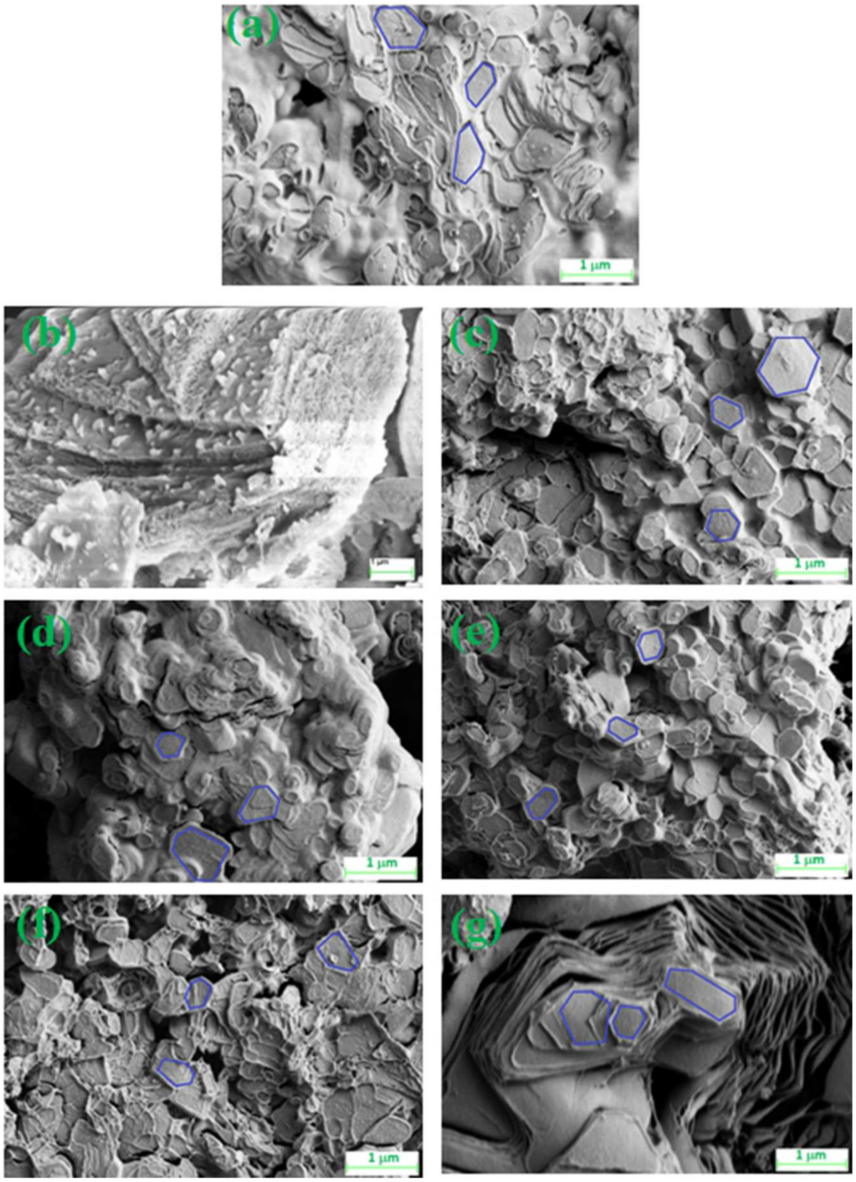

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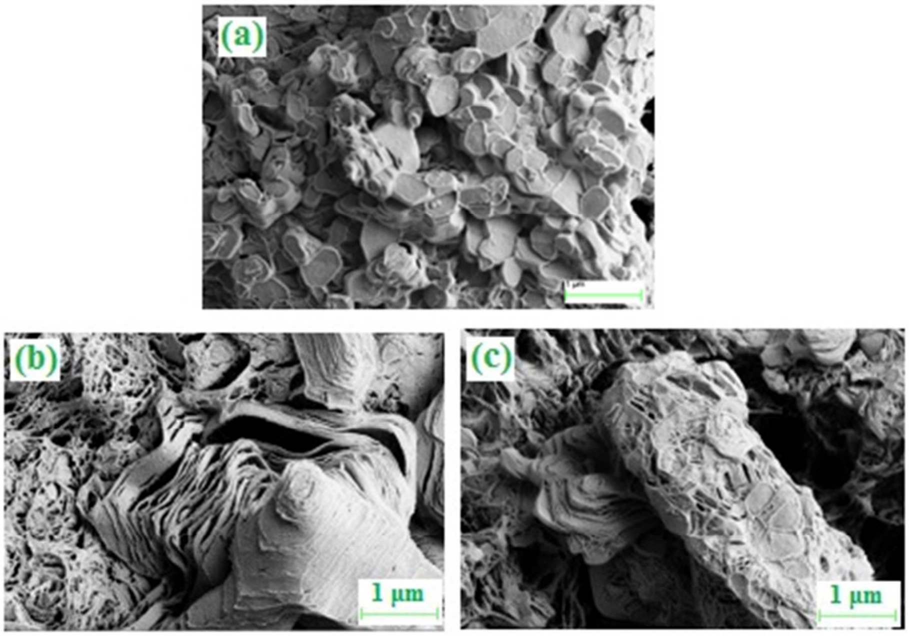

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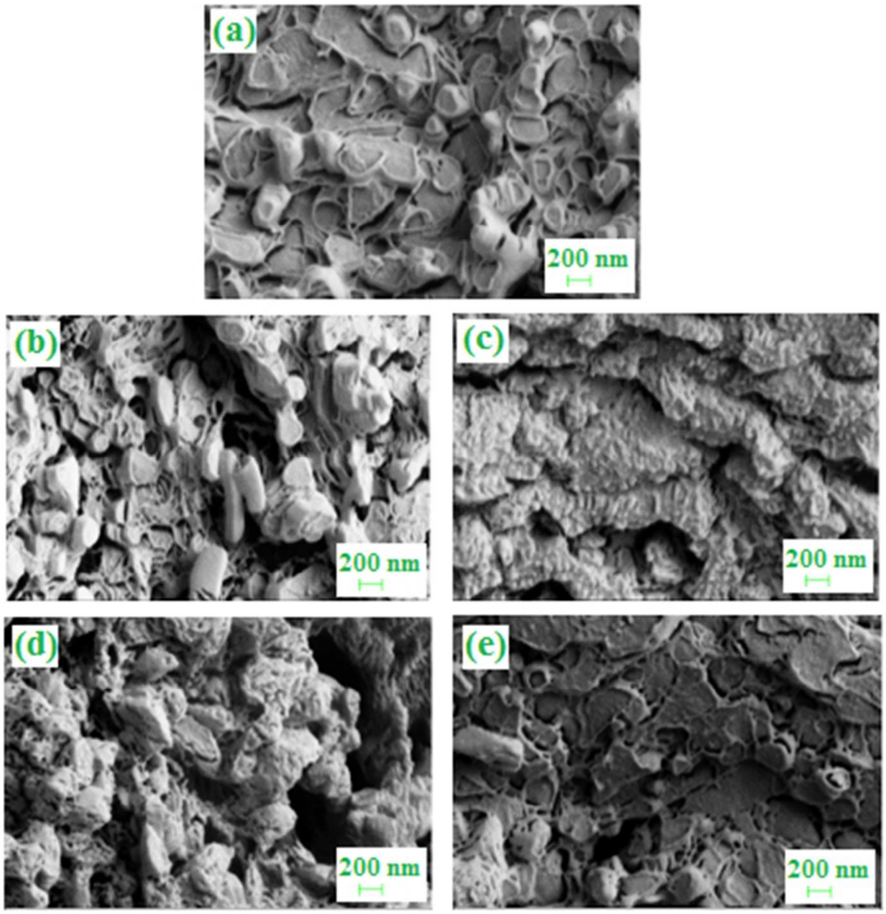

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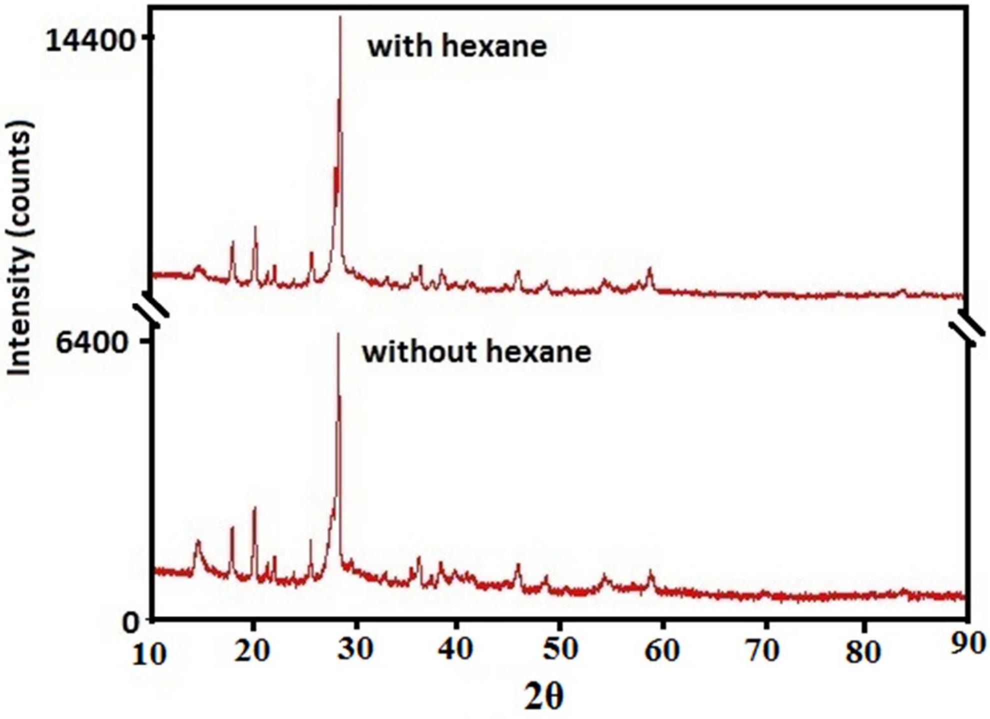

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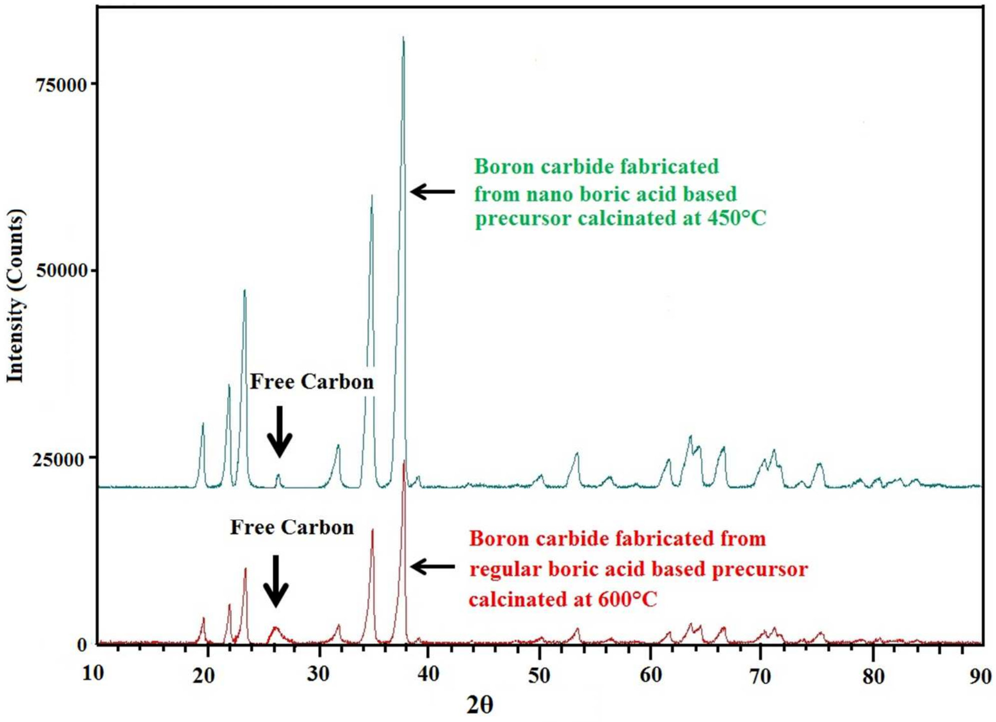

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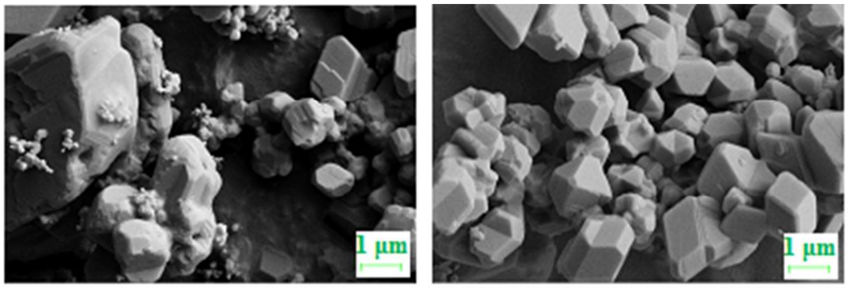

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