

CARBON NANO-NEEDLES PREPARATION BY USING SURFACTANTS AS CARBON SOURCE THROUGH IN SITU CARBONIZATION METHOD

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Abstract. Carbon nano-needles were successfully prepared using an in-situ carbonization method, employing the pores formed by the mixed crystallization of cesium chloride and sodium bromide as templates. The needle-like structures, resembling embroidery needles, were synthesized using three different surfactants as carbon sources: DTAB, SDBS and Tween-80. The resulting carbon nano-needles exhibited lengths ranging from 400-700 nm, 500-1000 nm and 2-4 μm , respectively. A series of characterizations, including SEM, FT-IR, XPS, XRD and Raman spectroscopy, confirmed that the prepared materials possess the fundamental characteristics of carbon nanomaterials. The successful synthesis of carbon nano-needles further demonstrates the feasibility of morphology modulation in carbon nanomaterials within a concentrated salt system, offering new methodologies and insights for the morphological control of carbon nanomaterials.

Keywords: Surfactants, carbon, nano-needles, alkali metal salts, crystallization, in situ carbonization.

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Received: 23 June 2025;

Accepted: 30 August 2025;

Published: 4 December 2025.

1. Introduction

Since the successful preparation of carbon nanocans by Iijima (1991), the modulation of morphology has been an important research direction. Arc discharge, chemical vapor deposition, templating, laser evaporation and catalytic reaction methods have been instrumental in the development of carbon nanomaterials (Iijima, 1991; Pradhan & Sharon, 2002; Konno *et al.*, 2004; Thess *et al.*, 1996; Liu *et al.*, 2000). Among these methods chemical vapor deposition is superior in regulating the morphology of carbon nanomaterials, chemical vapor deposition has successfully prepared a series of special morphologies such as octopus tentacle-like carbon nanostructures (Asif *et al.*, 2017), chrysanthemum-flower-like carbon nanomaterials (Ding *et al.*, 2016), carbon nanopears (Wang *et al.*, 2020), carbon nanocrosses (Dai & Deng, 2012), carbon nano-sheet powders (Yang *et al.*, 2008) and so on. Chemical vapor deposition has been instrumental in modulating carbon nanomorphology. However, the method also has certain shortcomings. For example, the preparation of carbon nanomaterials usually requires the passage of protective gas during the process and the gas consumption is large. It is not suitable for large-scale industrial production. Therefore, there is a need to explore a new method to modulate carbon nanomorphology.

During the previous explorations, special morphologies such as carbon nanoframes, carbon nanoblocks and nano-carbon octadecahedron were successfully prepared using the

interaction of surfactants and inorganic salts (Du *et al.*, 2022; 2024; Meng *et al.*, 2025). The successful preparation of these special shaped carbon nanomaterials proves that it is feasible to prepare carbon nanomaterials in concentrated salt system. This method can be further explored to prepare more special shaped carbon nanomaterials. Fortunately, a mixed salt was formed by co-crystallization of sodium bromide and cesium chloride during the exploration process. This mixed salt interacted with dodecyl trimethyl ammonium bromide (DTAB), sodium dodecyl benzene sulfonate (SDBS) and Tween-80, respectively, to successfully prepare needle-like carbon nanostructures at high temperatures. Compared with the preparation of nano-carbon octadecahedron in the concentrated salt system (Meng *et al.*, 2025), the successful preparation of carbon nano-needles (CNN) provides another new idea for the preparation of new carbon nanomaterials in the concentrated salt system. That is, two different types of inorganic salts were used to form new carbon nanomaterials by co-crystallization followed by interaction with surfactants. Compared with the templating method, this method offers distinct advantages: the templating method requires relatively hazardous solutions (e.g., hydrofluoric acid) for template removal (Jajcevic *et al.*, 2021; Puziy *et al.*, 2011; Konno *et al.*, 2004). In contrast, since inorganic salts are highly soluble in water, the inorganic salts can be easily removed after preparing carbon nanomaterials via the concentrated salt system. This novel idea and approach are an extension of the concentrated salt system and provides a preliminary basis for further exploration of the concentrated salt system.

2. Experiments and methods

Chemical reagents

DTAB ($C_{15}H_{34}BrN$, MW: 308.34, AR), SDBS ($C_{12}H_{25}NaO_3S$, MW: 272.38, AR), Tween-80 ($C_{24}H_{44}O_6$, MW: 428.6, AR), Cesium Chloride (AR) and Sodium Bromide (AR) for the experiments were of the brand name of Aladdin and were purchased at Compass platform (<https://www.shiyanjia.com>) for purchase. The above reagents were used directly without purification, in which the theoretical content of each element of surfactant is shown in Table 1. The experimental water was prepared in the laboratory.

Table 1. Theoretical content of each element of surfactant

Reagents	Theoretically calculated content of each element in the reagents (%)					
	C	N	O	S	Br	Na
DTAB	58.43	4.54	\	\	25.91	\
SDBS	52.92	\	17.62	11.77	\	8.44
Tween-80	67.26	\	22.40	\	\	\

Co-crystallization of cesium chloride with sodium bromide

To ensure that the two different inorganic salts were completely and fully mixed well, 121 g of sodium bromide and 1 g of cesium chloride were dissolved in 500 ml of deionized water to form a mixed solution. Two different types of inorganic salt solutions were crystallized by evaporative crystallization method and set aside. The morphology of cesium chloride and sodium bromide after crystallization is shown in Figure 1d.

Interactions of surfactants with mixed salts

5 ml of DTAB at a concentration of $1.776 \times 10^{-1} \text{ mol/L}$, $1.8 \times 10^{-2} \text{ mol/L}$ SDBS and $2.1 \times 10^{-1} \text{ mol/L}$ Tween-80, respectively, were spread flat in a 10 cm diameter petri dish

and the solution formed a thin film. Then, the prepared mixed salt solid was poured into a petri dish, making sure that the liquid film formed by the solution was completely covered and the petri dish was placed in a constant temperature drying oven at 90°C for adequate drying.

Preparation of carbon nano-needles (CNN)

The dried mixed salt solid from the previous step was poured into a crucible and placed in a muffle furnace, which was held for two hours after the temperature was increased to 600°C using a staged warming method. After the temperature of the muffle furnace was reduced to room temperature, the crucible was removed and the solid mixed salt was fully dissolved with deionized water. For the first time, the dissolved mixture was subjected to primary filtration with medium speed filter paper, a step aimed at removing larger carbons. Afterwards, the filtrate was secondly filtered with a sand core funnel and washed several times to obtain CNN. The filter membrane was dried in a constant temperature drying oven for subsequent characterization.

Characterization

In order to detect the properties of the prepared needle-like carbon nanomaterials, the characterization methods such as SEM (ZEISS Sigma 360, Germany), FT-IR (Thermo Fisher Scientific Nicolet iS20, USA), XRD (Bruker D8 Advance, Germany), Raman (Horiba LabRAM HR HR Evolution, Japan) and XPS (Thermo Scientific K-Alpha, USA) were used to analyse the morphology, elemental content, functionality and degree of graphitization. Evolution), Raman (Horiba LabRAM HR, Japan), XPS (Thermo Scientific K-Alpha, USA) and other characterization methods to analyse the morphology, elemental content, functional groups and graphitization degree of carbon nanomaterials.

3. Results and discussion

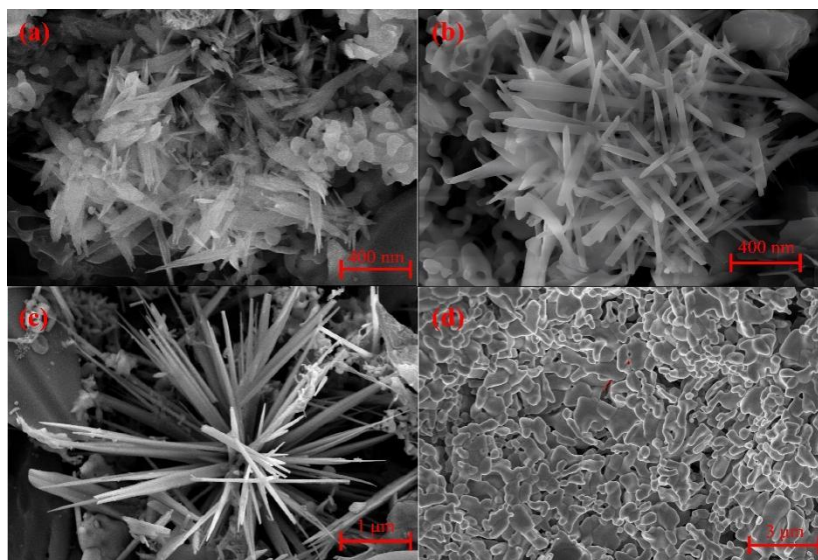


Figure 1. (a), (b), (c) carbon nano-needles by DTAB, SDBS, Tween-80, (d) Cesium chloride, sodium bromide co-crystal

SEM of the carbon nanomaterials was analyzed and the total lengths of the carbon nanomaterials prepared with three different surfactants respectively were 400 nm-700

nm, 500 nm -1000 and 2 μm -4 μm . That had a needle-sharp shape at one end. In response to the problem of high aggregation of carbon nano-needle. We believe that the carbon nano-needles do not form an aggregated form during the carbonization process, however, exist in the form of a single “needle”. In the process of removing inorganic salts, one “needle” naturally settles and aggregates together, which is shown as an aggregated form in SEM.

In the early stage, when carbon nanomaterials were prepared using a single inorganic salt in a concentrated salt system, the special acicular morphology did not appear (Du *et al.*, 2022; 2024; Meng *et al.*, 2025). In order to investigate the mechanism of carbon nano-needles formation, SEM tests were also performed on the crystalline salts of cesium chloride and sodium bromide and the results are shown in Figure 1d. Miraculously, the salt from the crystallization of the mixture of cesium chloride and sodium bromide showed a multi-hollow structure and also appeared to have a pore structure similar to the shape of carbon nano-needles. It is therefore reasonable to assume that it is the salt from the crystallization of cesium chloride mixed with sodium bromide that provides the template for the formation of CNNs.

The surfactant solution unfolds in the petri dish by surface tension and after the mixed crystalline salt is added to the surface dish, the surfactant solution is driven by capillary action into the cavity structure of the mixed salt. The solvent water is evaporated in a constant temperature drying oven and the surfactant is immobilized in the pores of the mixed salt. The special morphology of carbon nano-needles was later formed under high temperature carbonization. For the time being, this explanation is more reasonable for the formation of CNNs morphology.

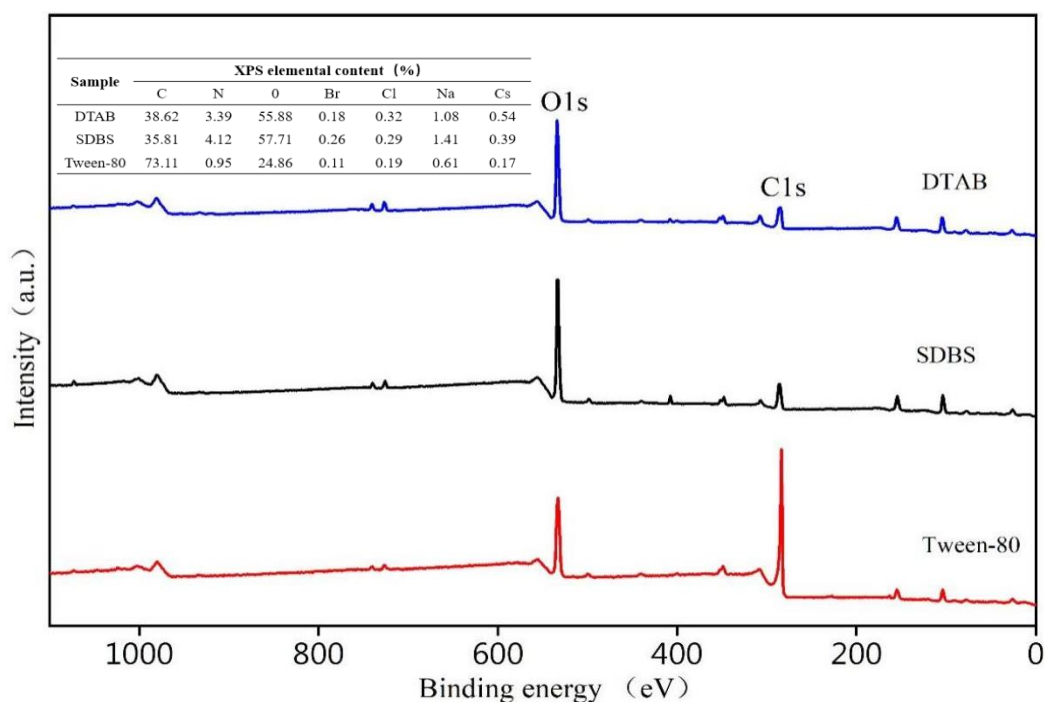


Figure 2. XPS mapping of carbon nano-needles

XPS is the main instrument for characterizing the chemical composition of materials and is often used in the characterization of carbon materials (Yamamoto, 2009; Zhang *et al.*, 2014; 2020; Okpalugo *et al.*, 2005). The prepared carbon nano-needles were subjected

to elemental analysis and the XPS patterns of the prepared CNNs are shown in Figure 2. In Figure 2, the XPS patterns and elemental compositions show that the three CNN contain basically the same elements, differing in their elemental content. The theoretical elemental content of the individual surfactants in Table 1 deviates somewhat from the charred elemental content in Figure 2. This is due to the fact that some elements of the surfactant in the charring process generate volatile small molecule gases, such as CO_2 , water vapor, etc.

In this process we also noted the content of the particular element N. SDBS and Tween-80 do not contain nitrogen by themselves, except for DTAB which contains nitrogen by itself. Surprisingly, the element N, which is least expected to be present in the carbide, was present in the carbide at a level far exceeding that of the impurity element in the AR-grade (99.7%) reagent. For this special phenomenon, we have conducted a preliminary analysis and there are three possible sources of nitrogen in the material. The first is that there is a certain error in XPS measurement or the material is contaminated during the test, resulting in a small amount of nitrogen in the XPS results of carbon nanomaterials. The second is the introduction of a small amount of nitrogen during the experimental operation. The third is that during the high-temperature carbonization process under air conditions, the co-crystallized sodium brom conditions, the co-crystallized sodium bromide and cesium chloride may undergo a nitrogen fixation reaction with nitrogen in the air, eventually leading to the presence of a small amount of nitrogen in the carbon nano-needles (CNNs). In this paper, only this special phenomenon is discovered. The specific causes of this phenomenon still need to be further verified and explained through a large number of experimental explorations in the future and thus this special phenomenon will not be discussed or demonstrated in detail in this paper.

Carbon nano-needles all contain elements such as Br, Cl, Na and Sc. During the preparation of carbon nanomaterials, the carbons were repeatedly and many times rinsed with deionized water and still contain these elements. We speculate that a very small portion of the salt is encapsulated by carbon nanomaterials during the carbonization process, resulting in elementally complex carbon nanomaterials.

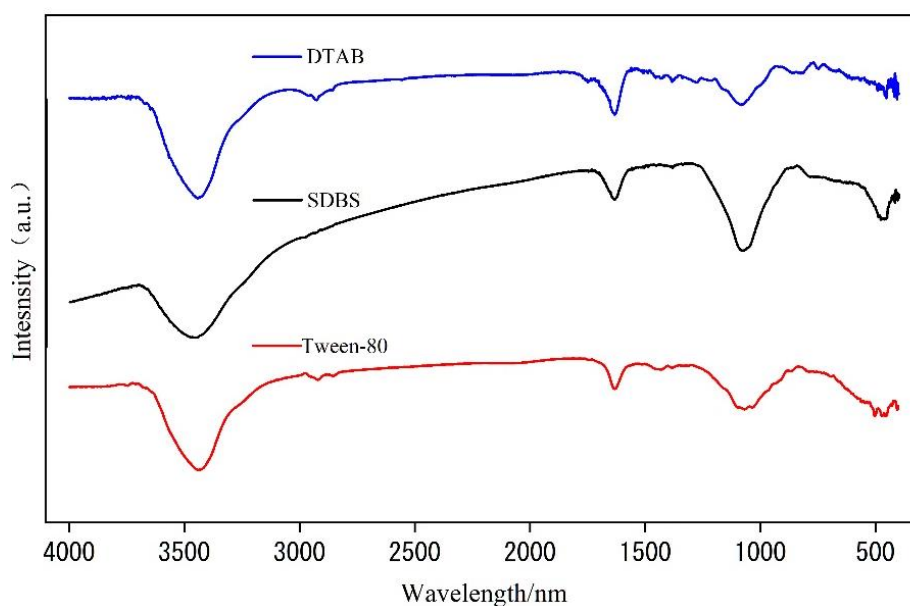


Figure 3. FT-IR pattern of carbon nano-needles

Fourier transform infrared (FT-IR) spectroscopy is widely used for the detection of functional groups in materials due to its advantages of high accuracy and sensitivity. Detection of functional groups on the surface of carbon nanomaterials is also often used (Zhang *et al.*, 2020; Gao *et al.*, 2022; Varga *et al.*, 2017; Hao *et al.*, 2024). Figure 3 shows the FT-IR patterns of carbon nano-needles, from which it can be seen that the FT-IR pattern curves of CNNs prepared by three different types of surfactants are approximately the same. It indicates that the prepared CNNs contain approximately the same functional groups. The peak appearing near $\sim 3500\text{ cm}^{-1}$ is often attributed to the stretching vibration of the O-H bond in the hydroxyl group (Liu *et al.*, 2020) and the probable reason for the appearance of this peak is that the carbon nanomaterials adsorb a portion of the moisture in the air during the detection process. The peak appearing near $\sim 2900\text{ cm}^{-1}$ is the stretching vibration of the C-H bond (Hao *et al.*, 2024); the peak appearing at $\sim 1600\text{ cm}^{-1}$ is attributed to the stretching vibration of the C=C bond (Liu *et al.*, 2019). The peak appearing between $1300\text{--}1000\text{ cm}^{-1}$ is the stretching vibration of the C-O bond (Jiang *et al.*, 2019). The peak arising between $800\text{--}700\text{ cm}^{-1}$ is the out-of-plane bending vibration of the C-C bond (Hao *et al.*, 2024). The small peaks occurring at $\sim 600\text{ cm}^{-1}$ are N-H bending vibrations (Zhao *et al.*, 2019; Dang *et al.*, 2019), which further suggests a small amount of nitrogen in the carbon material. The generation of peaks in the region smaller than 500 cm^{-1} is more complex and is not analyzed here as a priority.

The absorption peaks produced by infrared spectroscopy also indicate that during the high temperature carbonization of the three different types of surfactants in the concentrated salt system, the surfactants undergo high temperature cracking and other reactions to form new structures.

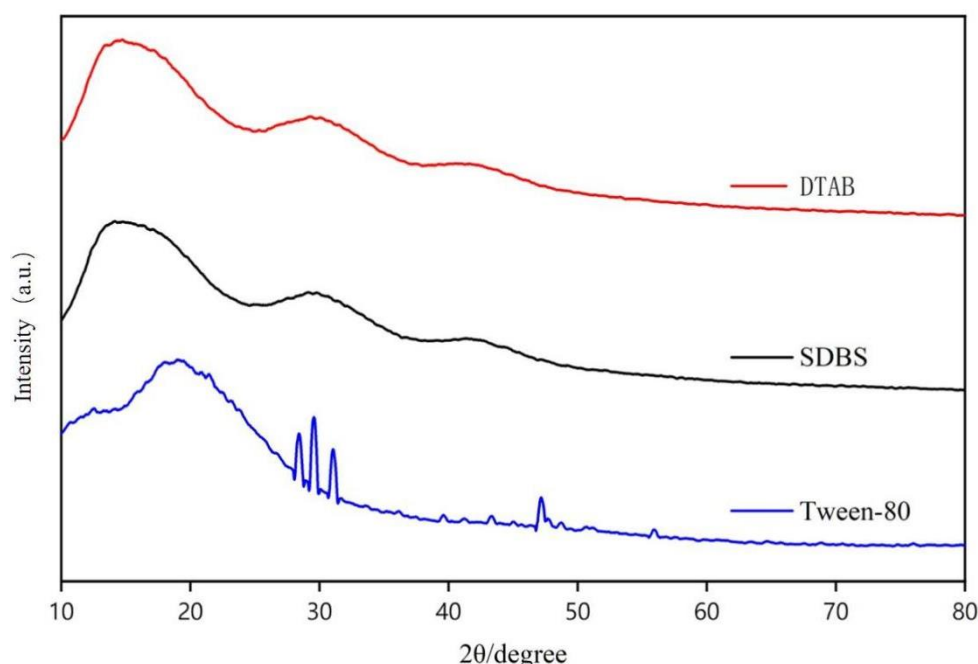


Figure 4. XRD pattern of carbon nano-needles

The XRD patterns of the prepared carbon nano-needles are shown in Figure 4 and the XRD patterns of the final carbon nano-needles prepared from the three different types of surfactants are approximately the same. The three CNNs also showed broad diffraction peaks between 10 and 20° and the appearance of such peaks indicates that all three carbon

nanomaterials have amorphous and amorphous structures (Ergun, 1968). The (002) crystallographic diffraction peak of the three carbon nanomaterials was shifted to the right ($\sim 30^\circ$) compared to the graphitic material with a 26.5° (002) crystallographic diffraction peak (Li *et al.*, 2005). This situation may have occurred during the high temperature carbonization process, where the carbon nanomaterials doped small amounts of inorganic salts. This affected the (002) crystal plane diffraction peaks of the carbon nanomaterials to be shifted. Tween-80 showed two diffraction peaks near 29° and 31° , which were probably caused by the introduction of some impurities during the experiment. All three carbon nanomaterials showed a weak (101) diffraction peak near 45.5° , which usually indicates disordered stacking and defects in carbon materials (Dandekar *et al.*, 1998). Tween-80 showed some sharper diffraction peaks around 55.85° , which were attributed to the presence of impurities introduced during the experiment.

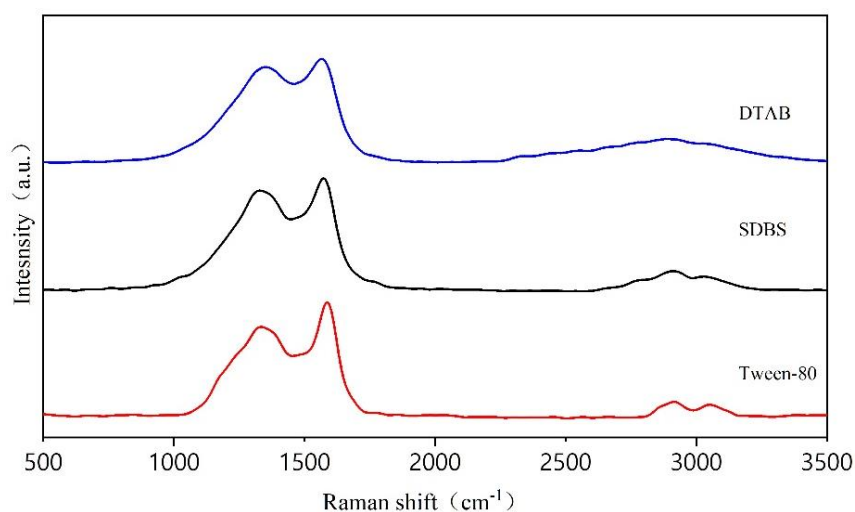


Figure 5. Raman map of carbon nano-needles

Raman spectroscopy is used as a classical method to characterize carbon nanomaterials. We also characterized the prepared carbon nano-needles by Raman spectroscopy. As can be seen from Figure 5, all three CNNs show D peaks generated by the respiration mode of sp^3 carbon and G peaks corresponding to the E_{2g} vibrational mode of graphite at 1300 cm^{-1} and 1580 cm^{-1} (Campos *et al.*, 2018; Madhurima *et al.*, 2024). The value of I_d/I_g indicates the degree of graphitization of the carbon nanomaterials and a smaller ratio of the two indicates a higher degree of graphitization. The I_d/I_g values of the three CNN are 0.92, 0.91 and 0.81, respectively and the higher values again indicate that the prepared CNNs are less graphitized and more defective.

In addition, the wider half peak widths of the D and G peaks of three carbon nano-needles are indicative of the lower crystallinity of the carbon nano-needles. This conclusion is also correlatively proved in the XRD patterns. We also noticed that the prepared CNNs also showed 2D peaks of weak intensity at 2690 cm^{-1} . These peaks are usually used to characterize the interlayer stacking pattern of carbon atoms in graphitic material samples (Madhurima *et al.*, 2024).

4. Conclusions

For the first time, carbon nano-needles were successfully prepared by using the pore structure generated by the mixed crystallization of different types of inorganic salts (cesium chloride and sodium bromide) as templates and different types of surfactants as the carbon source. The larger length-to-diameter ratios can be used to replace some of the carbon fibers in the future. By analyzing the XRD and Raman patterns of the three CNNs. It shows that the crystallinity and graphitization degree of the prepared CNNs are low and the degree of disorder and defects are high. The results of the characterization of the CNNs are comprehensively analyzed and the CNNs prepared under the concentrated salt system are not particularly 'perfect' from various perspectives, however, the role of the concentrated salt system in regulating the morphology of the carbon nanomaterials cannot be denied. The successful preparation of carbon nano-needles once again demonstrates the feasibility of the modulation of carbon nanomaterials in the concentrated salt system and provides new ideas and methods for the preparation of carbon nanomaterials in the future.

Acknowledgment

This work was supported by the Research Teams of Kunlun Scholars Project of Qinghai Province, China.

Disclosure Statement

The authors declare no conflict of interest.

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