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Research Article

Effect of Functionalized Carbon Nanotubes in the Detection of Benzene at Room Temperature

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In this paper, carbon nanotubes (CNTs) were functionalized by acid treatment and further functionalized with dodecylamine and were designated as CNT-carboxylic and CNT-amide, respectively. Then, functionalized CNTs produced were characterized with various methods to verify the attachment of a functional group. Performance of the functionalized CNTs in the detection of benzene gas was monitored at room temperature. The sample was dropped cast on the interdigitated transducer (IDT), and the changes in resistivity were recorded by a digital multimeter in a customized chamber under controlled humidity (~55%) environment. Based on the findings, it showed that the functionalized CNTs provide an extra active area for interaction between the gas analyte and CNTs, thus increasing their response and improving the sensitivity of the sensing material.

1. Introduction

Benzene, C_6H_6, is an important organic chemical compound that consists of six carbon atoms each bonded covalently with one hydrogen atom. One of the main uses of benzene is an intermediate in synthesizing other chemicals such as nitrobenzene, cumene, ethylbenzene, aniline, and cyclohexane. For example, ethylbenzene becomes a precursor of styrene, which is used in the production of polymers and plastics such as polystyrene (PS) and expanded polystyrene (EPS) [1]. Benzene has carcinogenic properties, which are well known for many years. United States Occupational Safety and Health Administration (U.S. OSHA) has set the limit of exposure in the workplace as 1 part per million (ppm) in 8-hour workday and 40-hour workweek [2]. U.S. OSHA has also fixed an action level of 0.5 ppm of benzene concentration in order to encourage lower exposure in the workplace [3].

Generally, benzene is detected using several techniques such as gas chromatography [4], mass spectrometry [5, 6], ion mobility spectrometry [7], and ultraviolet spectrophotometry [8, 9]. Even though the instruments are sensitive and produce results fast, they are bulky and not portable. Therefore, miniature gas sensors become the main interest in the detection of benzene [10–12] to overcome these obstacles. An excellent miniature gas sensor requires high sensitivity, fast response, high selectivity, and reproducibility [13]. Although sensing materials such as semiconductor metal oxides (SMO) have good performance, they need more power and have low selectivity.

Nanomaterials are a good candidate for sensing material because of their low power consumption and good chemical

selectivity [14]. Since its discovery by Ijima [15], carbon nanotubes (CNTs) have extensive application especially in the gas sensor field due to their nanosize and large surface area ratio which are important for the interaction between the gas analyte and CNTs. However, their uneven structure and agglomerations properties have limited their potential and make them less sensitive and selective [16]. Modification of the CNTs with functional groups [17], polymers [18], and metal oxide nanoparticles [19] is one of the several ways utilized to improve the compatibility of CNTs.

Modification of CNTs with various functional groups changes their electronic property, thus enhancing their selectivity and increasing their response towards specific gases. Noteworthy, the interaction of target molecules with a different functional group varies significantly [20]. Mainly, the CNTs is modified with a carboxylic group. The carboxylic group will create reactive sites at the sidewalls and end of the CNTs where vigorous interaction with target molecules happens. For example, it was shown [21] that single-walled carbon nanotubes attached with carboxylic (SWCNT-carboxylic) showed good repeatability and better response towards a mixture of 10 ppm carbon monoxide, CO, and ammonia, NH_3 gas. SWCNT-carboxylic demonstrated faster response towards CO than NH_3 gas. Other than that, the sensor synthesized from multiwalled CNTs functionalized with acid (MWCNT-carboxylic) was sensitive to hydrogen (H_2) gas with a detection limit of 0.05%, whereas pristine MWCNTs showed poor response to this gas [22]. The recovery time of the MWCNT-carboxylic sensor decreases to 100 s for 0.05% of H_2 gas as compared to 190 s for the pristine MWCNT. Leghrib et al. have developed a microsensor array for detection of benzene at room temperature by using plasma-treated multiwalled CNTs decorated with rhodium (Rh), palladium (Pd), gold (Au), or nickel (Ni). This sensor showed good sensitivity with the detection limit of benzene below 50 parts per billion (ppb) [10]. Also, a gas preconcentrator based on three different types of CNTs which is multiwalled , double-walled, and single-walled carbon nanotubes (MWCNTs, DWCNTs, and SWCNTs) was fabricated for efficient detection of benzene [23]. The results showed that MWCNTs produced via arc discharge displayed a good performance as the injector unit for the application in a gas preconcentrator.

In this paper, the functionalized CNTs were treated with sulphonitric mixture [24] and used as reaction precursor for amide functionalization by using dodecylamine as the functionalizing reactant. The surface morphology of the CNTs before and after functionalizing was examined by field emission-scanning electron microscopy attachment with energy dispersive X-Ray analysis (FESEM-EDX), and functional group attachment was confirmed by using Fourier Transform-Infrared spectroscopy (FT-IR). Then, the potential of the functionalized CNTs in the detection of benzene gas compared to the pristine CNTs and the effect of functionalization were studied by observing the changes in the resistivity of CNTs when exposed to benzene gas.

2. Methodology

2.1. Materials. Sulphuric acid (H_2SO_4, 98%), dodecylamine ($CH_3(CH_2)_{11}NH_2$, 99%), and nitric acid (HNO_3, 65%) were purchased from Merck Company (Germany). Carbon nanotubes (purity: 95%; type: multiwalled; inner diameter, ID: 5–10 nm) with a length of 10–30 μm were obtained from Nanostructured & Amorphous Materials, Inc (USA). All the chemical reagents were used without any purification and in analytical grade. For detection of benzene, benzene vapours in 100 ppm mixed with nitrogen gas were obtained from AGS Scientific Company (Singapore).

2.2. Carboxylic and Amide Functionalization of CNT. In a small beaker, 2.0 g of pristine CNTs was added to 3 : 1 H_2SO_4/HNO_3 (sulphonitric mixture) and sonicated with ultrasonication water bath for 2 hours at 70°C. After the treatment was done, the functionalized CNTs was filtered and washed repeatedly until the product reached pH 7 before drying for 24 hours in a vacuum oven [24]. This functionalized CNTs was labeled CNT-carboxylic.

For amide functionalization, dodecylamine was melted on a hotplate for half an hour at 80°C. As soon as the reactant melted fully, CNT-carboxylic was added and sonicated at the same temperature for a while before adding few drops of H_2SO_4 as a catalyst. The sonication process was continued for 5 hours. Finally, the product was filtered and washed several times until it reached pH 7 before drying for 24 hours in the vacuum oven. This functionalized CNTs was labeled CNT-amide. All the functionalized CNTs were characterized using FESEM-EDX and FT-IR analyses as well to ensure the favourable outcome in the attachment of carboxylic (-COOH) and amide (-$CONH_2$) functional groups on the CNTs.

2.3. Detection of Benzene by CNT. The pristine and functionalized CNTs were diluted in distilled water and sonicated for 30 minutes at 40°C. Then, by using a micropipette, 2.5 μL of the CNTs was dropped cast onto an interdigitated transducer (IDT) and dried for 30 minutes in the oven. Electrical contact was made by connecting two gold wires on the IDT and baked again in the oven for 30 minutes. IDT with the sample was placed in the customized chamber and connected to a digital multimeter to record the resistance changes. Benzene was injected 5 minutes alternately with nitrogen gas (as carrier gas) at the concentration from 0.125% to 1% at room temperature under controlled humidity environment (~55%). Resistance changes versus time were plotted to analyze the effect of functionalization of the CNTs on the detection of benzene. Other than that, the sensitivity of the CNTs also was calculated and plotted to compare the sensitivity of the pristine CNTs and the functionalized CNTs.

3. Results and Discussion

3.1. Characterization of Carboxylic and Amide Functionalization of CNT. Surface morphology and elemental composition of the functionalized CNTs were investigated by FESEM-EDX (JEOL 7600F; Institute of Bioscience, Universiti Putra Malaysia) with an accelerating voltage of 5.0 kV. Figures 1(a)–1(c) show the distinctive images of CNTs and

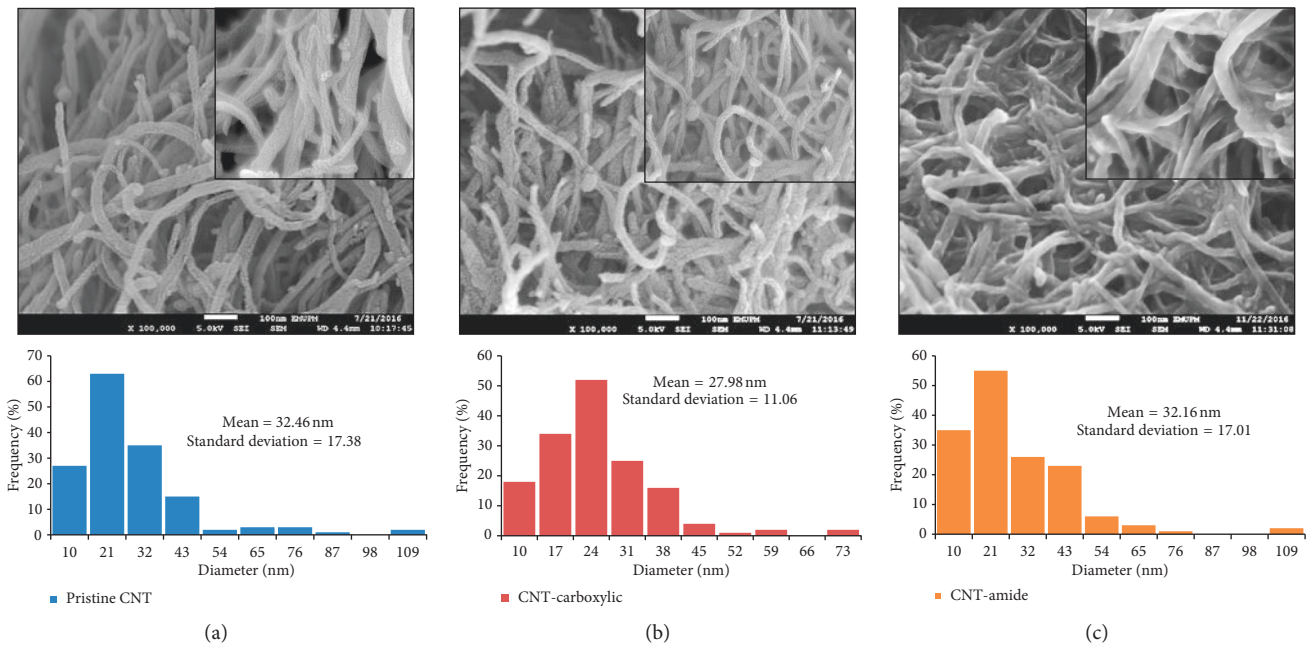


Figure 1: Micrographs of (a) pristine CNT, (b) CNT-carboxylic, and (c) CNT-amide.

their corresponding diameter size-distribution histogram. The pristine CNTs showed quite smooth structure compared to the functionalized CNTs. It is clearly shown in the insert images of Figures 1(a)–1(c) that the morphology of functionalized CNTs seems to be rough due to the treatment of acid during the functionalization process [25]. Diameter distribution of the pristine CNTs was in the range of 10 nm to 109 nm, and the mean diameter was 32.46 nm with a standard deviation of 17.38. Meanwhile, the functionalized CNTs are more aligned and dense caused by insertion of a new functional group [26]. Diameter distribution of CNT-carboxylic was decreased which was in the range of 10 to 73 nm, and the mean diameter was 27.98 nm with a standard deviation of 11.06, and diameter distribution of CNT-amide was slightly decreased which was in the range of 10 to 109 nm, and the mean diameter was 32.16 nm with a standard deviation of 17.01. The standard deviation of the functionalized CNTs samples was rather large, indicating that the diameter of the CNTs is inconsistent. In the functionalization process, the attachment of carboxylic (-COOH) and amide (-$CONH_2$) groups with the carbon-carbon double bond (C=C) of the CNTs depends on the stearic factor. Because of this effect, it is difficult to achieve a uniform functionalization; therefore, the diameter of the functionalized CNTs is inconsistent as expected. Thus, considering that the diameter alteration is caused by new functional groups [27], the elemental composition analysis has strengthened the result by showing an increase of oxygen element percentage and appearance of the nitrogen element (Table 1) as well.

The transformation produced by the insertion of new functional groups on the surface of the CNTs was qualitatively identified by using FT-IR spectroscopy (Figure 2). Table 2 shows the intensity of peaks appeared in the FT-IR spectra for all samples. All spectra showed bands at

Table 1: Elemental composition of pristine and functionalized CNTs.

Sample	Element (%)		
	C	O	N
Pristine CNT	97.02	2.98	—
CNT-carboxylic	92.84	7.19	—
CNT-amide	85.80	7.74	6.46

approximately 1640 cm^{-1} and 3100 cm^{-1}, which correlate with the C=C and O-H stretching vibrations, respectively [28]. The FT-IR spectrum of CNT-carboxylic showed the occurrence of a band at 1880 cm^{-1}, which is associated with the stretching of carbonyl from the carboxylic acid group. Moreover, an increase in the intensity of the band at 2880 cm^{-1} compared with the pristine CNTs spectrum confirmed the attachment of the carboxylic acid group (Table 2). According to a previous study [29], the hydroxyl and carboxyl groups that attached during the modification of CNTs with sulphonitric mixture were contributed to this increment. After CNT-carboxylic was further functionalized with an amine, bands at 1650, 1404, and 3300 cm^{-1} were discovered in the spectrum, which may be designated to C=O (amides), N-H bending, and N-H stretching, respectively [30]. Thus, the FT-IR spectrum of CNT-amide confirms that the surface of CNT-carboxylic was accomplished, treated by the amide functional groups.

3.2. Detection of Benzene by CNT. Figure 3 shows resistance changes of CNTs when exposed to benzene gas with different concentrations at room temperature. As reported by Leghrib et al. [31], functionalized CNTs have good response towards benzene. Basically, detection of benzene is related to transfer of electron charges between the gas analyte and sensing materials,

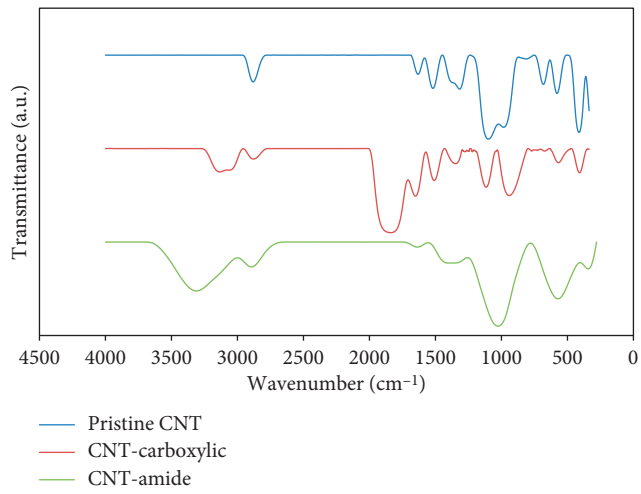
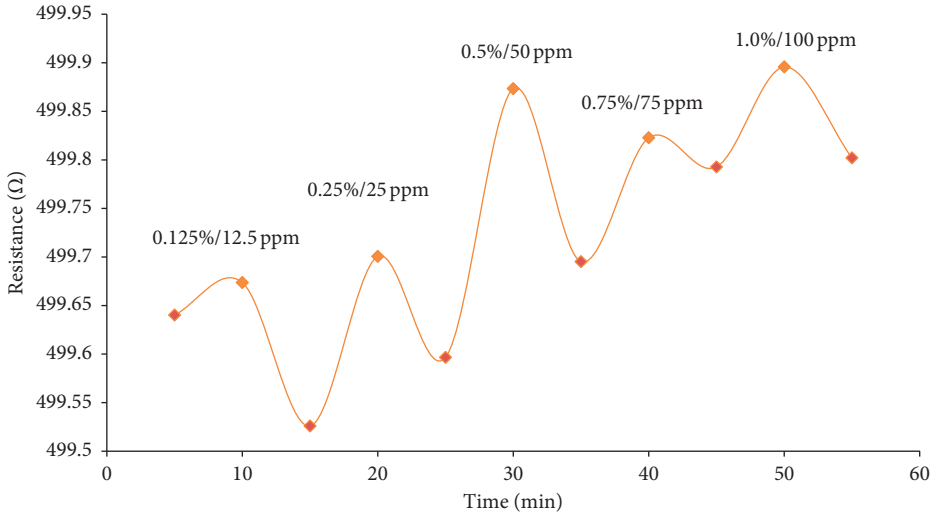


Figure 2: FT-IR spectra of (a) pristine CNT, (b) CNT-carboxylic, and (c) CNT-amide.

Table 2: Intensity of peaks appeared in pristine CNT, CNT-carboxylic, and CNT-amide.

Sample	Peak (cm^{-1}) (bond)	Intensity (%)
Pristine CNT	1640 (C=C)	88
	2880 (C=O carboxyl)	74
CNT-carboxylic	1640 (C=C)	54
	1880 (C=O carbonyl)	12
	3100 (O-H)	78
	2880 (C=O carboxyl)	89
CNT-amide	1650 (C=O amide)	98
	1404 (N-H bending)	79
	2880 (C=O carboxyl)	81
	3300 (N-H stretching)	57



(a)

Figure 3: Continued.

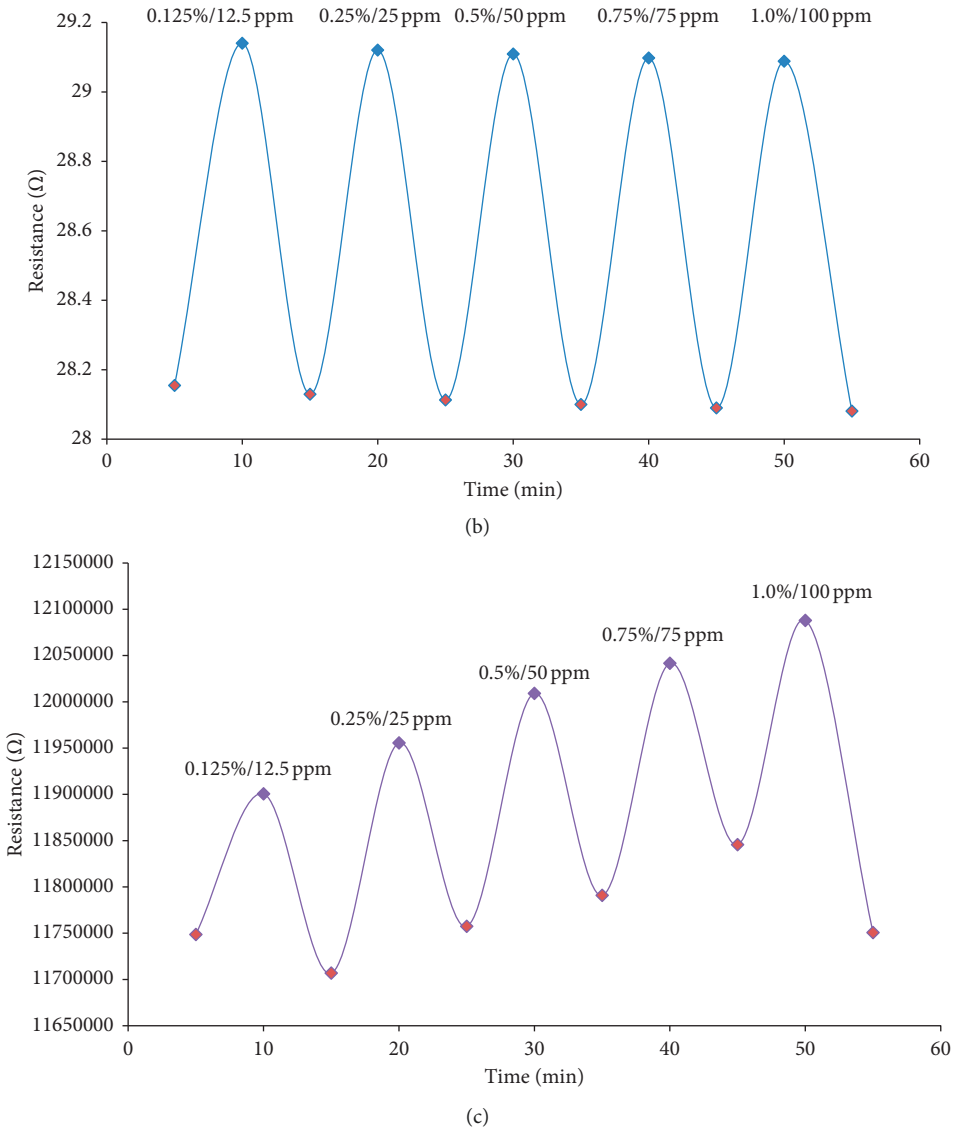


Figure 3: Resistance changes of (a) pristine CNT, (b) CNT-carboxylic, and (c) CNT-amide towards benzene gas at room temperature.

in our case, CNTs. As it can be seen, the reciprocal changes of resistance are directly proportional to increasing concentration of benzene gas. The pristine CNTs as a sensing material showed unstable resistance reading upon exposure to benzene gas. Meanwhile, the functionalized CNTs showed the better response when increasing the benzene gas concentration.

The resistance of CNT-carboxylic increases when exposed to benzene gas, and after nitrogen gas is purged back into the system, the baseline resistance is regained. This is due to the interaction with benzene gas that shifts the Fermi level of the CNTs away from the valence band, resulting in an increase of resistance of the CNTs [32]. Because of the attachment of the carboxylic acid group on the surface of CNTs, the gas analyte most likely attached to the tail of the carboxylic group [23], which prevents the gas analyte to reach the surface of the CNTs, resulting in the same value of resistance when exposed to different concentrations of benzene gas. But, for CNT-amide, the resistance of benzene gas increased when the concentration increased. This is because the saturation effect exists when the molecule is not desorbed completely on the surface of CNTs at room temperature unlike in the case of CNT-carboxylic. Thus, the baseline resistance of CNT-amide was difficult to regain upon the cleaning phase.

Moreover, CNTs with the functional group acts as an extra active area for gas adsorption [33], allowing more vapours to interact and eventually releasing more number of free electrons [12]. The transfer of free electron to the conduction band of oxide groups (carboxylic and amide), in the functionalized CNTs changing the hole concentration. These electrons shuttled into the nanotubes network due to positioning of tubes and the functional group. Such a process creates hole-electron recombination and reduce the number of holes in the tube. As a result, the resistance of CNTs increased as well as their sensitivity upon exposure to the benzene vapours.

Figure 4 shows a graph of the sensitivity of CNTs upon exposure to benzene gas. Sensitivity (S) of pristine CNTs and functionalized CNTs was estimated by Equation (1) [34],

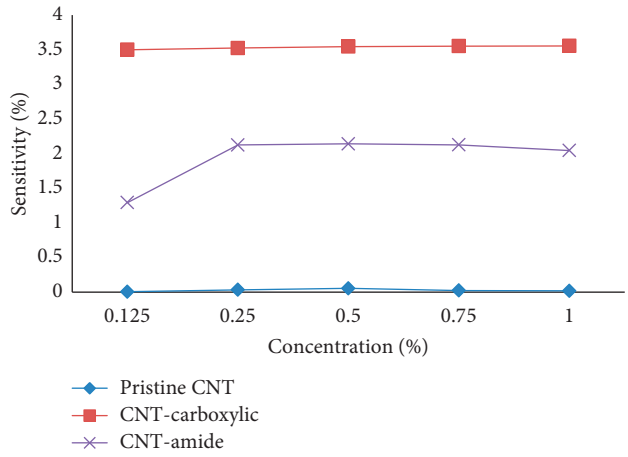


Figure 4: Sensitivity of the CNT upon exposure to benzene gas.

where R_g is the resistance of CNTs when exposed to benzene gas and R_o is the resistance of CNTs when exposed to nitrogen gas:

$$S = \frac{R_g - R_o}{R_o} \times 100\%. \quad (1)$$

From the graph, it is clearly seen that functionalized CNTs showed higher sensitivity for every concentration of benzene gas than the pristine CNTs; for example, at 0.125%, the sensitivity of the pristine CNTs is only 0.0067% compared to CNT-carboxylic (3.4995%) and CNT-amide (1.2929%). Based on this result, it can be concluded that the functionalized CNTs was highly responsive towards benzene gas due to the occurrences of a functional group on the surface of the CNTs [35].

4. Conclusion

The CNTs was functionalized with the carboxylic and amide functional groups. From the characterization analysis, it was confirmed that the functional group successfully attached on the surface of the CNTs. The functionalized CNTs shows better response and high sensitivity towards benzene at room temperature. Thus, it gives a huge impact in the detection of other gas using functionalized CNTs, a sensing material with high sensitivity and low production cost.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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