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# Synthesis of carbon nanofibres from waste chicken fat for field electron emission applications



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## ABSTRACT

Carbon nanofibres (CNFs) with sea urchin-like morphology were synthesised from waste chicken fat precursor via catalytic thermal chemical vapour deposition method at 750 °C. The CNFs showed amorphous structures under high-resolution transmission electron microscopy, micro-Raman spectroscopy and X-ray diffraction examination. X-ray photoelectron spectroscopy analysis confirmed that the core of the sea urchin-like CNFs was composed of Fe<sub>3</sub>C formed within the first 20 min of synthesis time. The growth of amorphous CNFs from agglomerated Fe<sub>3</sub>C particles was favourable due to the high heating rate applied during the synthesis. Field electron emission examination of the CNFs indicated turn-on and threshold field values of 5.4 and 6.6 V  $\mu$ m<sup>-1</sup> at current density of 1 and 10  $\mu$ A cm<sup>-2</sup>, respectively. This study demonstrates that waste chicken fat, a low-cost and readily available resource, can be used as an inexpensive carbon source for the production of CNFs with a potential application in field electron emitters.

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# 1. Introduction

Fibrous sp<sup>2</sup>-based carbon systems are of tremendous interest due to their potential applications in field emission devices [1], electrodes in supercapacitors [2], hydrogen storage [3], and sensors [4]. Carbon nanofibres (CNFs) exhibit cylindrical shapes similar to those of carbon nanotubes (CNTs), but the former have a larger diameter of approximately 100–500 nm [5] as well as different structural and textural characteristics. In general, CNFs are more disordered, with the graphitic crystals less oriented than in the CNTs. CNFs can be classified into the high and low graphitic categories [2]. High-resolution transmission electron microscopy (HR-TEM) and micro-Raman analysis investigations of the in-plane graphitic crystallite size have shown that the low graphitic CNFs have an amorphous character.

Conventional CNF synthesis uses non-renewable, petroleumbased precursors such as methane and acetylene [6]. Because of

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increasing environmental concerns, alternative precursors obtained from environmentally friendly and renewable sources such as camphor and turpentine from vegetable oil have been explored [1,7]. This effort has now been extended to chicken fat, which is the waste product of the poultry industry. Chicken fat cannot be adaptively reused in other sectors such as animal food production [8] because of the high content of contaminants and free fatty acids (FFA). Recent reports [9-11] on the composition of chicken fat (Table 1) shows that the major FFA in chicken fat are  $oleic(C_{18}H_{34}O_2)$ , palmitic $(C_{16}H_{32}O_2)$ , and linoleic  $(C_{18}H_{32}O_2)$  acids. As revealed by gas chromatography mass spectrometry (GC-MS) analysis (Table S1 in Supplementary material), these FFA consist of long hydrocarbon chains. The rich hydrocarbon content is favourable for the use of chicken fat as a carbon precursor for the CNFs production. The selection of chicken fat as the carbon precursor is also cost-effective because it is a free carbon source. This method of CNF production also reduces the environmental impact due to the improper disposal of chicken fat, which contributes to the land and water pollution [12]. Moreover, current management practices of food waste such as incineration are unsatisfactory [13] due to the resulting greenhouse gas emissions, which contribute to global climate change.

Therefore, we propose utilising waste chicken fat as an alternative and green carbon source for the production of CNFs,

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Table 1Fatty acid composition of chicken fat.

Reference	Fatty acid composition (wt%)				
	Palmitic	Palmitoleic	Stearic	Oleic	Linoleic
[9]	24.00	5.80	5.80	38.20	23.80
[10]	19.82	3.06	6.09	37.62	31.59
[11]	28.36	5.75	6.70	41.12	14.32

which can in turn be used as the carbon precursor for the production of vertically aligned CNTs, as reported previously [12,14]. To the best of our knowledge, this is the first report of the synthesis of CNFs from waste chicken fat using a simple, viable, and cost-effective thermal chemical vapour deposition (TCVD) method.

#### 2. Materials and method

Chicken oil was extracted from chicken fat and skin by a dry rendering process. In this process, the fat and skin were heated to 200 °C to separate liquid oil from solids. The oil was recovered by filtration and then mixed directly with the ferrocene at 5.33 wt%. The oil-catalyst mixture was stirred thoroughly for 30 min. 6 ml of the mixture was vapourised at 470 °C in vapourisation furnace and synthesis was carried out at 750°C for 60 min. Other synthesis process was similar to that previously reported [12]. The heating rate applied in this study was increased to 150 °C min<sup>-1</sup>, in contrast to the 2°C min<sup>-1</sup> heating rate used in the previous work. The product formed on the silicon substrate was collected and characterised using field emission scanning electron microscopy (FESEM-Hitachi SU8020), HR-TEM (Tecnai G2 20S Twin), micro-Raman spectroscopy (Renishaw InVia microRaman System, 514 nm laser), X-ray diffraction (XRD-Bruker AXS D9), and X-ray photoelectron Spectrocopy (XPS-Omicron Dar400). The field electron emission (FEE) measurement was performed at  $3 \times 10^{-5}$  Pa in a diode configuration. The area of the electron emission was 0.1 cm<sup>2</sup>, and the separation between electrodes was  $100 \,\mu$ m.

#### 3. Results and discussion

Fig. 1(a-e) shows the FESEM images of the CNF sample that was synthesised from the waste chicken fat. As is evident from the images in Fig. 1(a-c), the synthesised CNFs were in the form of sea urchin-like microstructures. These clusters consist of a nucleus with multiple needle-like nanostructures grown radially from the nucleus, with a diameter of 145-150 nm. The higher magnification FESEM image shown in Fig. 1d reveals the smooth surface of the needle-like nanostructures with blunt tips. Interestingly, spiralshaped structures with smaller diameters of approximately 100-120 nm as well as coiled needle-like structures were also present in some parts of the sample (Fig. 1e). The HR-TEM image (Fig. 1f) reveals that the CNFs have a solid-core structure (i.e. without an internal hollow). No crystalline structure is present because the graphitic layers are not well-defined, demonstrating the amorphous nature of the synthesised CNFs. Fig. 1g indicates that the growth of the CNFs proceeded via a tip growth mechanism, where most of the oval-shaped catalyst particles resided on the tip of the CNFs and were smaller than the diameter of the CNFs.

The micro-Raman spectrum of the as-grown CNFs is shown in Fig. 2(a) and consists of two broad bands at 1342 and 1603 cm<sup>-1</sup>. These bands were assigned to the D- and G-bands that were attributed to the disorder or defects in the graphite crystal and the stretching mode of the C—C bond in the graphite plane, respectively. The obtained spectrum can be ascribed to amorphous carbon because the D-band was broad and the full width at half maximum (FWHM) of the G-band was 57.6 cm<sup>-1</sup>, which was twice

the size of the FWHM for the band of the high graphitisation carbon fibres  $(23 \text{ cm}^{-1})$  [15]. The peaks for this sample are sufficiently broad, such that there is a significant overlap between the bands [16]. Moreover, the G-band of the synthesised CNFs shifted to a higher wavenumber (1603 cm<sup>-1</sup>) relative to the G-band of graphitic carbon, which is normally located at 1580 cm<sup>-1</sup> [9,17-20]. The difference is attributed to the less-ordered nature of the carbon and low graphitisation [21]. The value of the  $I_D/I_C$  ratio that represents the degree of CNFs' crystallinity was 0.79: this ratio depends on the in-plane graphitic crystallite size  $(L_a)$ .  $L_a$  is calculated using the Knight formula:  $L_a$  (nm) =  $C \times (I_D/I_G)^{-1}$ , where the coefficient C = 44 Å is only applicable for  $\lambda_L$  = 514 nm [22]. The degree of graphitisation is higher for the larger value of  $L_a$  [16]. The calculated value of  $L_a$  in this study was 5.5 nm, which is consistent with previous work [2,15,16], where  $L_a$  was found to be  $\sim 5$  nm. This result was in contrast to the typical 9–11 nm  $L_a$  value for crystalline CNFs obtained using the catalytic CVD method [16,22]. The small  $L_{a}$ value found in the present study confirms the amorphous nature of the sample.

The amorphous nature of the CNFs sample was also verified by the existence of a weak XRD graphite peak with a broad FWHM at  $26^{\circ}$  (Fig. 2(b)) that can be attributed to the small  $L_a$  value of the sample. The presence of other phases in the sample was apparent in the XRD pattern; based on the peaks at  $30.1^{\circ}$ ,  $56.6^{\circ}$ , and  $61.9^{\circ}$ , these were identified as Fe<sub>3</sub>C at  $46.3^{\circ}$  and Fe<sub>3</sub>O<sub>4</sub> that resulted from the spontaneous oxidation of the iron particles when the sample was handled in air [23].

In order to measure the state of Fe catalyst during the synthesis process. XPS analyses were performed on the samples grown at various period of synthesis time starting with lower synthesis time of 20 min. Then followed by samples synthesised at 40 and 60 min, respectively. The C1s core level spectra of these samples are shown in Fig. 3(a)-(c). The main peak at about 284.9, 284.8 and 284.9 eV which was assigned to  $C-C \operatorname{sp}^2$  hybrids as well as peak located at 288 eV that corresponded to C=O were identifiable for the entire samples. Meanwhile, the peak at about 283.5 eV that corresponded to Fe<sub>3</sub>C was only detected in the sample grown for 20 min. The increasing amount of amorphous carbon in the samples with further increase of decomposition time of carbon precursor makes the iron phases undetectable by XPS [24]. Meanwhile, the Fe2p spectra of the samples (Fig. 3(d)) shows two peaks located at about 711 and 724 eV attributed to  $Fe2p_{3/2}$  and  $Fe2p_{1/2}$ , respectively. The presences of these peaks further affirmed the existence of Fe<sub>3</sub>O<sub>4</sub> [25] as detected in XRD analysis. Therefore, it can be deduced that the formation of Fe<sub>3</sub>C which later formed the core of sea urchinlike CNFs has started as early as 20 min synthesis time. A small quantity of Fe<sub>3</sub>C relative to the thickening of amorphous carbon as synthesis time increases to 40 and 60 min would be the reason for the absence of the Fe<sub>3</sub>C peak in XPS spectrum [24,26].

The possible mechanisms of the CNFs' synthesis are shown in Fig. 4. Initially, ferrocene was thermally decomposed at 190 °C [27] into the transition metal, hydrogen and hydrocarbon species [28]. The nucleation of Fe or Fe–C particles in Fig. 4A(i) (see path 1) was similar to that proposed by Boi et al. [23]. However, some modifications were made here because of the amorphous nature of the produced CNFs. The carbon molecules from the decomposition of ferrocene dissolved into the Fe particles with the resultant diffusion gradient [23] forming Fe<sub>3</sub>C. This process occurs in the first 20 min of synthesis time as confirmed by the XPS analysis. When the saturation was reached, the carbon condensed out, encapsulating the Fe<sub>3</sub>C particles (Fig. 4A(ii)). We suggest that the entire process occurs in the vapour phase. Due to the high heating rate (150°C min<sup>-1</sup>) applied in the system instead of the low heating rate  $(2 \circ C \min^{-1})$  applied previously [12], the particle transport rate was high, which promoted the aggregation of the Fe<sub>3</sub>C structure. This structure formed a large, spherically shaped nucleus, as shown in



Fig. 1. (a-e) FESEM and (f and g) HR-TEM images of the CNFs synthesised using waste chicken fat.

Fig. 4B. As the temperature increased to 470 °C, chicken oil was fully decomposed into the lighter hydrocarbon and other vapour elements  $(C_xH_yO_{Z(1)} \rightarrow C_x,H_{y,O_{Z(2)}} + C_x,H_{y,N(g)} + CO_{(g)} + CO_{2(g)} + H_2O_{(1)} + OH^{\bullet}_{(g)}$  where x = 2x' + 2, y = 2y' + 3 and z = z' + 5) and continuously dissolved in the aggregated Fe<sub>3</sub>C cluster. Once the Fe<sub>3</sub>C cluster was supersaturated with carbon, the CNFs structures

elongated radially from the nucleus. This was initiated by a spherically symmetric diffusion gradient [23]. As synthesis time increased, the amorphous carbon has thickened causes  $Fe_3C$  can no longer detected by XPS.

Another possible nucleation of the sea urchin-like structure (see path 2) was proposed; here, the aggregation of the Fe particles



Fig. 2. (a) Micro-Raman spectrum and (b) XRD pattern of the CNFs grown from waste chicken fat.

occurred prior to the formation of  $Fe_3C$  particles. Initially, Fe particles from decomposition of ferrocene deposited on Si substrate (Fig. 4B(i)). High heating rate applied caused high particle transport rate, thereby resulting in agglomeration of Fe particles (Fig. 4B(ii)). The hydrocarbon molecules from the decomposed ferrocene and the precursor subsequently underwent decomposition and dissolved into the Fe particles forming large, spherically shaped  $Fe_3C$  nucleus. This process followed by saturation and the outward diffusion of carbon to form the CNFs from the nucleus (Fig. 4C(iii) and D). A tip growth model has been

proposed for the synthesis of sea urchin-like CNFs due to the presence of the oxidised Fe particles ( $Fe_3O_4$ ) in the sample, as detected by the XRD and XPS measurement.

Fig. 5 displays the typical emission current density versus applied electric field (*J*–*E*) curve and the corresponding Fowler Nordheim (F–N) plot for the CNFs grown from chicken fat. From the curve, the observed turn-on and threshold were approximately 5.4 and 6.6 V  $\mu$ m<sup>-1</sup>, corresponding to current densities of 1 and 10  $\mu$ A cm<sup>-2</sup>, respectively. These values were comparable to those obtained using the sample synthesised from waste cooking palm



Fig. 3. XPS photoelectron spectra of C1s for sample synthesised within (a) 20, (b) 40, and (c) 60 min and (d) Fe2p for 20-60 min samples.



**Fig. 4.** The possible mechanism for the formation of sea urchin-like CNFs. Path 1-A(i): Nucleation of Fe or Fe–C particles. A(ii): Encapsulation of Fe<sub>3</sub>C by carbon that condensed out. B: Spherically shaped nucleus formed by the aggregation of the Fe<sub>3</sub>C structure promoted by high heating rate applied. Path 2–C(i): Fe particles from ferrocene decomposition deposited on Si substrate. C(ii): Fe particles agglomerated due to high heating rate applied. C(iii): Decomposition and dissolution reactions of the hydrocarbon molecules from the decomposed ferrocene and the precursor forming large, spherically-shape Fe<sub>3</sub>C nucleus. D: Outward diffusion of carbon to form the CNFs from the nucleus.



Fig. 5. J-E curves and corresponding F-N plots (inset) for the synthesised CNFs.

oil (turn-on and threshold field values of 6.2 and 7.7 V  $\mu m^{-1}$ , respectively) [29]. The effective field enhancement factor ( $\beta$ ) calculated from the slope of the F–N plot was 821. These results indicate that CNFs synthesised using waste chicken fat have the potential to be used as emitters in electron emission devices.

# 4. Conclusions

CNFs with a sea urchin-like structure were successfully synthesised from waste chicken fat for the first time by the TCVD method. This was achieved by using a high precursor heating rate of  $150 \,^{\circ}\mathrm{C\,min^{-1}}$ . The method is simple and may be scaled up for industrial use. The radial structures of sea urchin-like CNFs consisted of solid-core fibres departing from a central nucleus. The central nucleus contains aggregated Fe<sub>3</sub>C particles that formed within the first 20 min of synthesis time. We proposed that the growth of the sea urchin-like structure was due to the large aggregation of the ferrum species resulting from the high rate of particle transport that is driven by the high precursor heating rate.

The FEE properties show that the synthesised CNFs have a potential for use in field electron emitters.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j. materresbull.2015.04.068.

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