

## Materials science communication

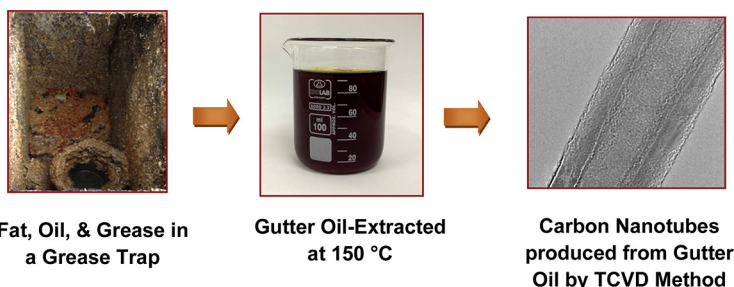
## Synthesis, structural, and field electron emission properties of quasi-aligned carbon nanotubes from gutter oil

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## HIGHLIGHTS

- Gutter oil was used as starting material to synthesise CNTs by TCVD method.
- CNTs of good quality ( $I_D/I_G \sim 0.66$  and purity  $\sim 81\%$ ) were successfully produced.
- The synthesised CNTs show a potential for field electron emission application.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Quasi-aligned carbon nanotubes (CNTs) have been successfully synthesised from the simple pyrolysis of gutter oil as starting material and ferrocene as a catalyst. The synthesis process was performed at synthesis and vaporisation temperatures of 800 and 250 °C, respectively, in a thermal chemical vapour deposition furnace. The CNTs synthesised using gutter oil have an overall diameter of about 30–50 nm, length of 30 μm,  $I_D/I_G$  ratio of 0.66, and purity of 81%, comparable to those obtained using conventional carbon sources. A field electron emission study of the CNTs exhibited a low turn-on and threshold field of 1.94 and 2.94 V μm<sup>-1</sup>, which corresponded to current densities of 100 μA cm<sup>-2</sup> and 1.0 mA cm<sup>-2</sup>, respectively which indicate that the CNTs synthesised are suitable candidates for use as field electron emitters. The synthesised CNTs from gutter oil also open up potential mass production applications in energy storage devices. This study demonstrates that gutter oil, a low-cost and readily available resource, can be used as an inexpensive carbon source for the mass production of CNTs.

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## 1. Introduction

In recent years, a great deal of research effort has been devoted to the production of carbon nanotubes (CNTs) using low cost and readily available carbon precursor from waste resources such as waste plastic [1], scrap tyre rubber [2], waste glycerol [3], and waste engine oil [4]. These efforts have arisen due to the high cost of petroleum-based precursors (such as methane [5], acetylene [6],

and ethylene [7] and [8]) as the supplies are predicted to be depleting in next few decades. Prior to the use of waste-based material as an alternative source of carbon, crop-based carbon sources including camphor [9–11], turpentine [12], eucalyptus [13], palm [14–17], coconut, olive, and corn oil [18] have been utilised to synthesise CNTs. Although the use of these green and renewable carbon sources seems to be appealing, their use for carbon precursors would lead to food shortage, increase the price of food products, and may contribute to environmental damage as they are grown on land converted from rainforests, peatlands, savannas, and grasslands [19]. These problems indicate that the crop-based oils may not be the only future resources for the production of CNTs.

Previous studies have shown that food-based waste such as waste cooking palm oil [20] and waste chicken fat [21–23] have been utilised for the synthesis of CNTs. These syntheses are possible due to the high carbon content originating from the fatty acids contained in the structures of both the oil and the fat. The utilisation of food-based waste precursor can now extend to gutter oil extracted from fat, oil, and grease (FOG) deposited in sewerage system. Gutter oil is a promising carbon source as it consists of a mixture of carbon-rich hydrocarbons. The gas chromatography mass spectrometry (GC-MS) and CHNS analyses in Table 1 and Table 2, respectively confirm that gutter oil has high carbon content and low C:H ratio, ideal to produce good-quality CNTs due to the low probability of by-product formation such as amorphous carbon [21]. In addition, the presence of oxygen in the structure will oxidise the amorphous carbon in-situ [24]. The high level of carbon content in the gutter oil is the main contributing factor that makes gutter oil the suitable precursor for mass production of CNTs.

The utilisation of gutter oil as carbon source not only can reduce the production cost of CNTs, but also help to manage the disposal problem of FOG. Conventional treatment of FOG such as land filling, land application, and incineration are not economical and associated with soil and air pollution problem. Alternatively, FOG are recycled for various uses including biodiesel [25], biogas [26] and

**Table 2**  
CHNS elemental analysis of gutter oil.

Element	C	H	N	S
Weight%	69.774	10.277	3.976	0.043

also sold as cooking oil at below-market rates [27]. Although recycling is considered to be eco-friendly, there is a significant technical challenge in converting the FOG to biodiesel where the traditional base catalysis does not work well with grease due to the formation of soap between the base and free fatty acid in FOG [25]. Meanwhile, converting the gutter oil into cooking oil can be hazardous and even fatal to human health as it contains large quantities of bacteria and other dirty elements that congregate in sewage and can cause diarrhoea as well as other serious health effects. Gutter oil is also highly toxic as it contains harmful substances such as fatty and trans fatty acids as well as strong carcinogens such as aflatoxins, which accumulate as a result of several uses before disposal. The fatty acids in gutter oil however can be useful in the production of carbon material, including CNTs.

These factors indicate that the waste from improper disposal of FOG is a potential source for CNT production. To the best of our knowledge, this is the first attempt to use gutter oil in the synthesis of CNTs by thermal chemical vapour deposition (TCVD) method. The potential of the CNTs produced from gutter oil as a field electron emission (FEE) cathode and electrode material for supercapacitor devices were also studied.

## 2. Materials & method

Gutter oil is an extract of waste FOG collected from a grease trap of a food service establishment located in a sewerage system. FOG was heated to 150 °C to extract the oil with dark brown in colour. The extracted oil was filtered to remove debris. The EDS analysis of the gutter oil (Fig. 1 (a)) shows the presence of C, O and metallic components of Na and Ca. The high weight % of C in gutter oil is consistent with the CHNS analysis (Table 2). The detail analysis of the gutter oil using inductively coupled plasma-optical emission spectrometry (ICP-OES) was carried out to support the presence of metallic components performed by EDS. The measurement was repeated for three times to obtain the average value of the metallic components. In contrast to EDS, Fe and K were also detected together with Ca and Na. However, the concentrations of Ca, Na, Fe, and K were found to be relatively lower than the values reported by others [28] (Table 3). The low metal concentration detected in the FOG was due to the origin of the FOG. The FOG used in this study was collected from restaurant, which the amount of metal components were known to be low as they came from the food grease trap resource. The Na in the gutter oil originated from the cooking salt (NaCl) used in the cooking process [20] while Ca was presumably released from the concrete surfaces under low pH conditions [29]. In order to synthesise CNTs, 5.33 wt% ferrocene was added to the gutter oil extracted and mixed thoroughly for 30 min. The CNT synthesis process followed a technique reported previously [21]. Briefly, a two-stage tube furnace was used, where the gutter oil-ferrocene mixture was placed in one stage and substrates of *p*-type (100) silicon in the other. The synthesis temperature was set at 800 °C and the vaporisation temperature was set at 250 °C according to the vaporisation temperature of the gutter oil, determined previously using thermogravimetric analysis (TGA) (Fig. 1 (b)). The CNTs samples produced were characterised using field emission scanning electron microscopy (FESEM-JEOL JSM 7001F), high resolution transmission electron microscopy (HR-TEM-JEOL JEM 2100F), micro-Raman spectroscopy (JASCO-NRS 1500W), and

**Table 1**  
Compounds identified and yield (% area) by GC-MS of gutter oil.

Compounds	Retention time	Area (%)
Aromatic hydrocarbons		
Ethylbenzene	3.380	1.027
Total		1.027
Aliphatic hydrocarbons		
2-methyloctacosane	20.073	1.061
2-methyloctacosane	20.155	2.253
Tetratetracontane	20.266	3.023
Octadecanamide	21.053	0.861
Tetratetracontane	23.267	1.185
Total		8.383
Oxygenated hydrocarbons		
2-Pentanone, 4-hydroxy-4-methyl-	3.077	2.655
Tetrahydrofuran, 2,2-dimethyl-	3.864	4.031
2-Pentanone, 3-methyl-	4.114	2.258
Hydroperoxide, 1-ethylbutyl	4.376	0.777
Hydroperoxide, 1-methylpentyl	4.505	0.722
Cyclopentane, 1-acetyl-1,2-epoxy-	4.740	2.727
Octanoic acid	7.534	1.511
Nonanoic acid	8.918	1.791
Dodecanoic acid	12.766	1.807
Pentadecanoic acid	15.050	2.299
n-Hexadecanoic acid	17.171	62.580
Oleic acid	18.849	1.708
Octadecanoic acid	19.036	1.842
Total		86.708
Nitrogenated hydrocarbons		
Hexadecanamide	19.269	1.173
9-Octadecenamide, (Z)-	20.871	2.710
Total		3.883

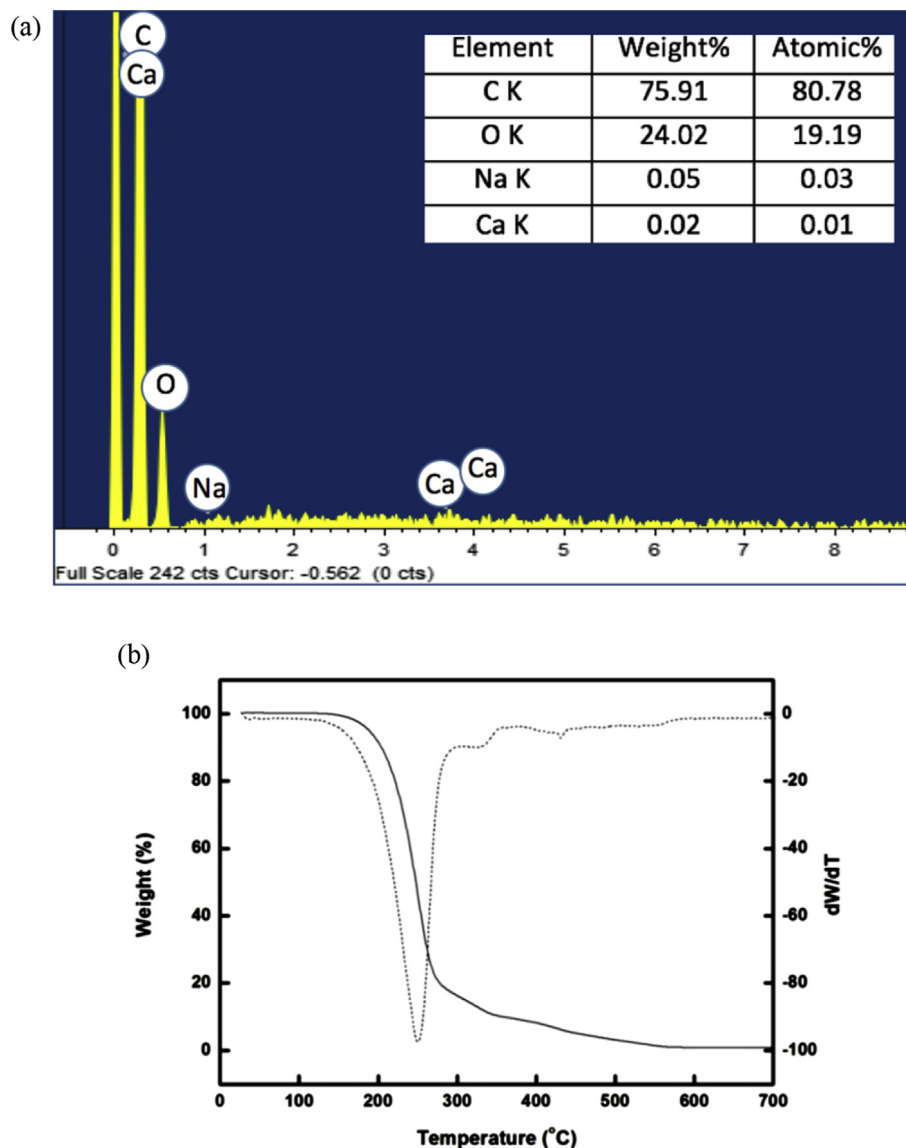


Fig. 1. (a) EDS and (b) TGA analysis of gutter oil.

Table 3

Concentrations of metallic components in gutter oil analysed by ICP-OES.

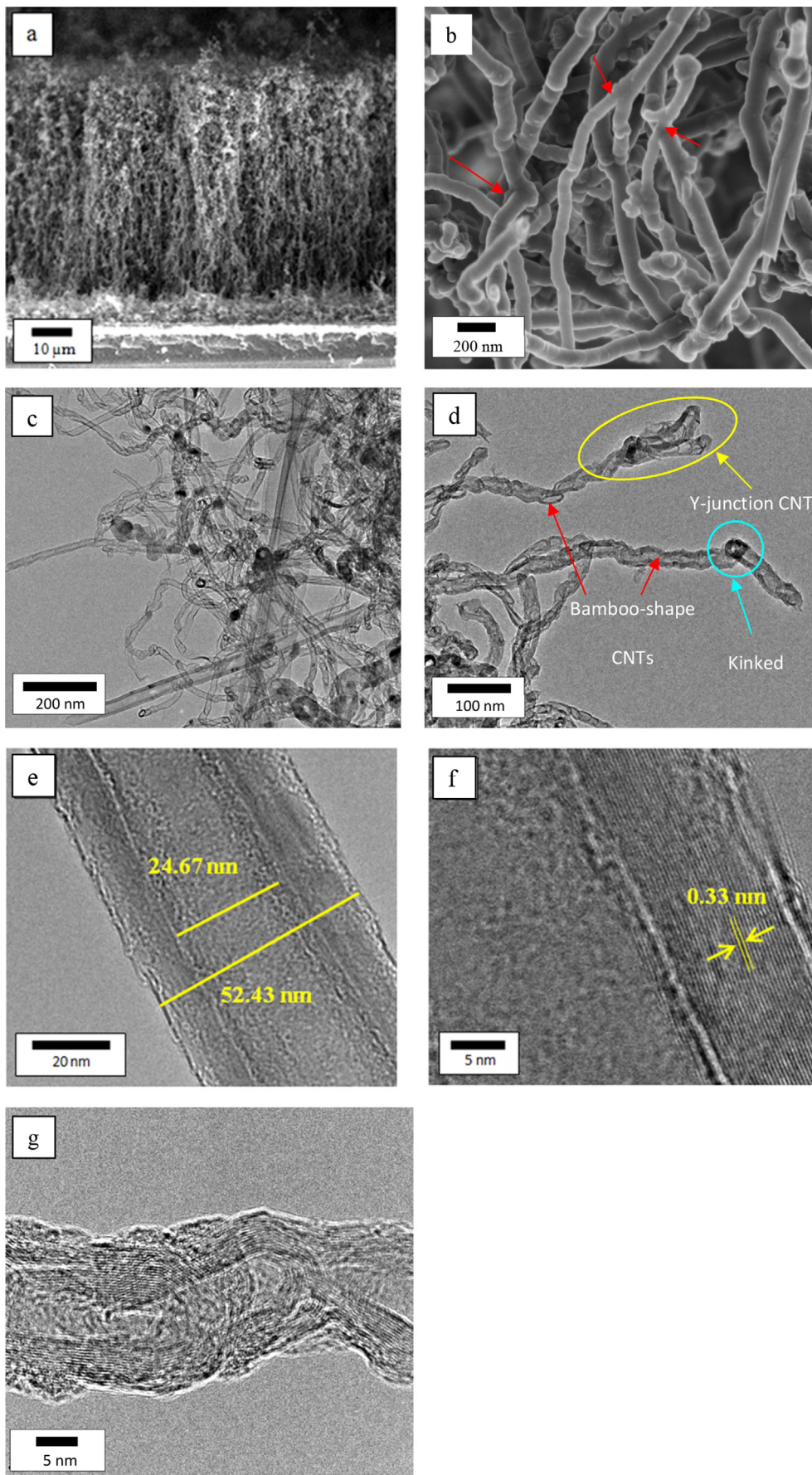
Metal	Mean concentration (mg/L)	Reported range of samples tested (mg/L) [2]
Ca	0.227	240–37,359
Fe	0.056	70–577
K	0.080	14–11,968
Na	0.297	34–54,348

TGA (Mettler Toledo SDTA 851). Field emission measurements were performed at  $3 \times 10^{-5}$  Pa in a diode configuration. The area of the electron emission was  $0.1 \text{ cm}^2$  and separation between electrodes was  $100 \text{ }\mu\text{m}$ . Cyclic-voltammometry (CV) measurements were conducted on the sample using a Gamry Potentiostat Series-G750 to study the potential application of synthesised CNTs for supercapacitor devices. Two films with cross-sectioned area of  $1 \text{ cm}^2$  obtained from the synthesised CNTs samples were used as electrode materials. The films were sandwiched between a polymeric electrolyte separator [30]. The CV measurement was conducted at a

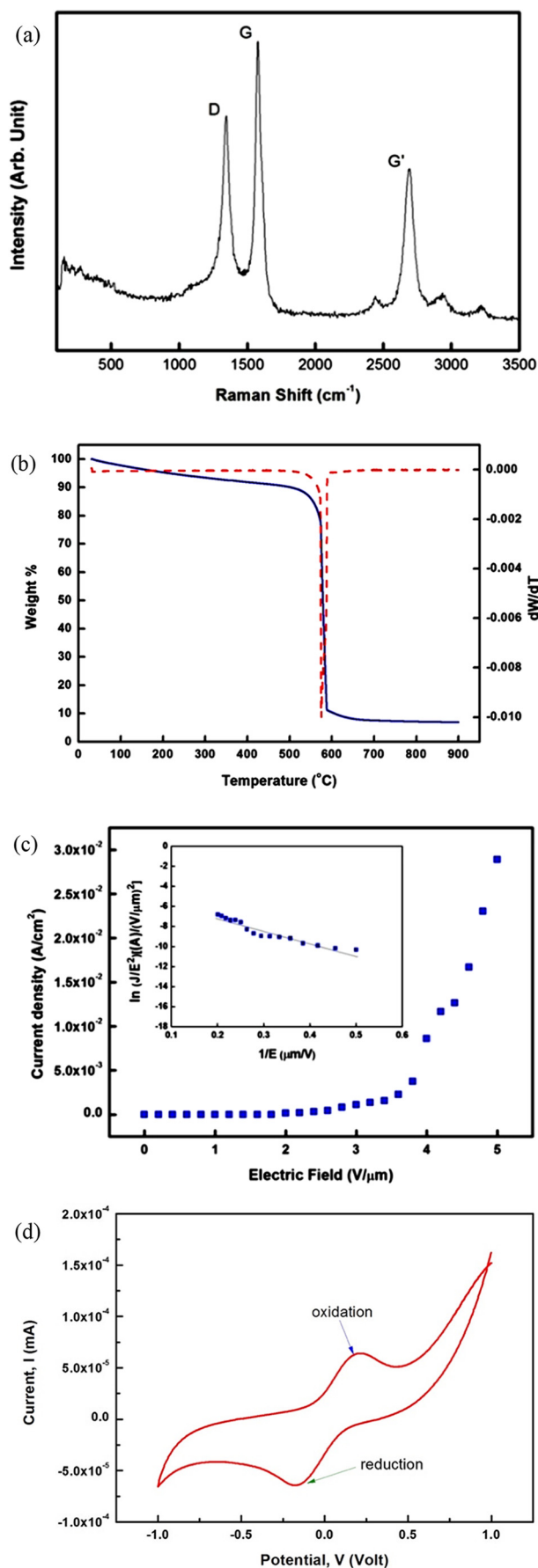
potential window of  $-1.0$  to  $1.0 \text{ V}$  with the scan rate of  $100 \text{ mV/s}$ .

### 3. Results & discussions

Fig. 2 (a)–(b) show the FESEM images of the CNTs synthesised using gutter oil. Quasi-aligned CNTs with the length of about  $30 \text{ }\mu\text{m}$  were observed, giving an average growth rate of about  $0.50 \text{ }\mu\text{m min}^{-1}$ . Higher magnification FESEM image in Fig. 2 (b) shows that the synthesised CNTs have average diameters of  $30\text{--}50 \text{ nm}$ . The morphology also consists of non-tubular carbon



**Fig. 2.** (a)–(b) FESEM image of CNTs from gutter oil in different magnification, (c) lower magnification TEM image of CNTs, (d) kinked, Y-junction and bamboo-shape CNTs, and HR-TEM images of (e)–(f) hollow-centred and (g) bamboo-shape CNT.



structures. The presence of Y-junction and branch CNTs were observed as indicated by the arrows in Fig. 2 (b). The presence of sulphur in the gutter oil presumably initiates the formation of the Y-junction CNTs [31]. Sulphur was also reported to enhance the catalytic activity of iron catalyst as well as enhancing the metal filling of CNTs [32].

The hollow-centred structures shown in Fig. 2 (c) confirm the formation of CNTs. In addition, the TEM image reveals the CNTs morphology also consists of kinked, Y-junction, and bamboo-shape CNTs (Fig. 2 (d)). The HR-TEM image (Fig. 2 (e)) shows that the outer and inner diameters of the hollow-centred CNTs were 52.4 and 24.7 nm, respectively. Higher magnification image in Fig. 2 (f) reveals that the as-grown CNTs were multi-walled CNTs contained approximately 42 graphitic layers with interlayer spaces of ~0.33 nm, consistent with the interplanar spacing of graphite (0.3354 nm) [33]. Meanwhile, the HRTEM image of the bamboo-shape CNTs (Fig. 2 (g)) shows the hollow compartments separated by curved carbon domes from the graphitic sheets joining the tubes inner walls.

The micro-Raman spectrum of the CNTs shown in Fig. 3 (a) yielded the D, G and G' peaks commonly associated with graphitic materials. The sharp G peak indicates the formation of well-graphitised CNTs. This result is in good agreement with the HR-TEM result. The  $I_D/I_G$  ratio that represents the degree of crystallinity of the CNTs was found to be 0.66 indicating that the synthesised CNTs have a reasonable crystallinity. This value is comparable to those values reported for CNTs synthesised using other waste-based precursors such as waste plastic ( $I_D/I_G \sim 0.63$ ) [1], waste glycerol ( $I_D/I_G \sim 0.77$ ) [3], waste cooking palm oil ( $I_D/I_G \sim 0.64$ ) [20], waste chicken fat ( $I_D/I_G \sim 0.63$ ) [21], and waste engine oil ( $I_D/I_G \sim 0.90$ ) [4]. The high value of D and G' peaks observed in micro-Raman spectrum might be attributed to the presence of bamboo-shape CNTs. This is in agreement with the results previously reported by [34–36] of bamboo-shape CNTs that showed high value of D and G' peaks and the value of  $I_D/I_G$  ratio of 1.18, 0.82 and 1.05 respectively.

The TGA and differential TGA (DTGA) curves of the CNTs are shown in the Fig. 3 (b). A weight loss about 3.1% was detected at a temperature of 110–250 °C which is attributed to the combustion of hydrocarbon impurities. The weight loss of around 8.2% which occurred at 250–550 °C was mainly decomposition of amorphous carbon. The oxidation of CNTs started at 508 °C followed with the combustion that occurred at 575 °C as indicated by the exothermic peak shown in DTGA curve. The combustion of CNTs ended at 683 °C leaving a 7.7% remnant that is associated with the catalyst residue and other non-volatile constituents. The purity of the CNTs synthesised from gutter oil was found to be 81%. This demonstrates that high purity CNTs can be produced from waste-based precursor.

Fig. 3 (c) displays the typical emission current density versus applied electric field ( $J-E$ ) curve and the corresponding Fowler–Nordheim (F–N) plot of the CNTs. From the  $J-E$  curve, the observed turn-on and threshold field were approximately 1.94 and 2.94  $V \mu m^{-1}$  which corresponded to current densities of 100  $\mu A cm^{-2}$  and 1.0  $mA cm^{-2}$ , respectively. The maximum current density was found to be 30  $mA cm^{-2}$ . This value is comparable to those obtained from the CNTs synthesised using conventional [34,37,38], green [39], and waste carbon precursors [4,20] as well as the CNTs-metal oxide composite [40–42] (Table 4).

The inset figure shows the F–N plot that was obtained according to the typical F–N equation as shown in Eq. (1):

Fig. 3. (a) Micro-Raman spectrum, (b) TGA analysis, (c)  $J-E$  curve with corresponding F–N plot as an inset image, and (d) CV curve of CNTs synthesised using gutter oil.

**Table 4**  
Turn-on field and maximum current density of CNTs and CNTs-metal oxides samples.

Turn-on field	Max. current density	Reference
0.65 V $\mu\text{m}^{-1}$ at 100 $\mu\text{A cm}^{-2}$	14.5 mA $\text{cm}^{-2}$	CNTs from $\text{C}_2\text{H}_2$ precursor: Kar et al., 2015 [34]
2.20 V $\mu\text{m}^{-1}$ at 10 $\mu\text{A cm}^{-2}$	100 mA $\text{cm}^{-2}$	CNTs from $\text{C}_2\text{H}_4$ precursor: Sridhar et al., 2014 [37]
0.33 V $\mu\text{m}^{-1}$ at 10 $\mu\text{A cm}^{-2}$	16 mA $\text{cm}^{-2}$	CNTs from acetone precursor: Lee et al., 2012 [38]
2.80 V $\mu\text{m}^{-1}$ at 0.01 mA $\text{cm}^{-2}$	1.2 mA $\text{cm}^{-2}$	CNTs from camphor oil precursor: Asli et al., 2013 [39]
2.25 V $\mu\text{m}^{-1}$ at 10 $\mu\text{A cm}^{-2}$	6.0 mA $\text{cm}^{-2}$	CNTs from waste cooking palm oil precursor: Suriani, Roslan, & Rusop, 2010 [20]
4.10 V $\mu\text{m}^{-1}$ at 0.1 $\mu\text{A cm}^{-2}$	3.7 $\mu\text{A cm}^{-2}$	CNTs from waste engine oil precursor: Suriani et al., 2015 [4]
1.94 V $\mu\text{m}^{-1}$ at 100 $\mu\text{A cm}^{-2}$	30.0 mA $\text{cm}^{-2}$	This work
1.21 V $\mu\text{m}^{-1}$ at 10 $\mu\text{A cm}^{-2}$	1.2 mA $\text{cm}^{-2}$	CNTs- $\text{Fe}_2\text{O}_3$ composite: Guo et al., 2013 [40]
0.38 V $\mu\text{m}^{-1}$ at 100 $\mu\text{A cm}^{-2}$	2.4 mA $\text{cm}^{-2}$	CNTs- $\text{TiO}_2$ composite: Xu et al., 2015 [41]
0.75 V $\mu\text{m}^{-1}$ at 10 $\mu\text{A cm}^{-2}$	0.5 mA $\text{cm}^{-2}$	CNTs-CuO composite: Hu et al., 2015 [42]

$$\ln(J/E^2) = \ln(A\beta^2/\phi) - B\phi^{3/2}/\beta E \quad (1)$$

where  $J$  is the current density,  $E$  is the applied field, and  $\phi$  is the work function (5 eV for carbon),  $A$  and  $B$  are constants ( $A = 1.54 \times 10^{-6} \text{ A eV V}^{-2}$  and  $B = 6.83 \times 10^9 \text{ eV}^{-3/2} \text{ V/m}$ ) and  $\beta$  is the field enhancement factor which can be calculated using the slope of the linear part of the F–N plot. A linear curve was exhibited, which is in agreement with the F–N equation, and thus confirms that the observed current is generated by field emitted electrons. The effective  $\beta$  calculated for the sample was 6158. The field emission results obtained indicated that the CNTs synthesised using gutter oil are a good candidate for field electron emission. This corresponds to the low turn-on field, high maximum current density and high  $\beta$  value. In addition, the entangled CNTs secure a good electrical contact all across the CNTs [43], and have better long-term stability than the straight CNTs that are easily deflected upon high-field application [44]. Furthermore, the HR-TEM and micro-Raman spectroscopy indicated that the synthesised CNTs

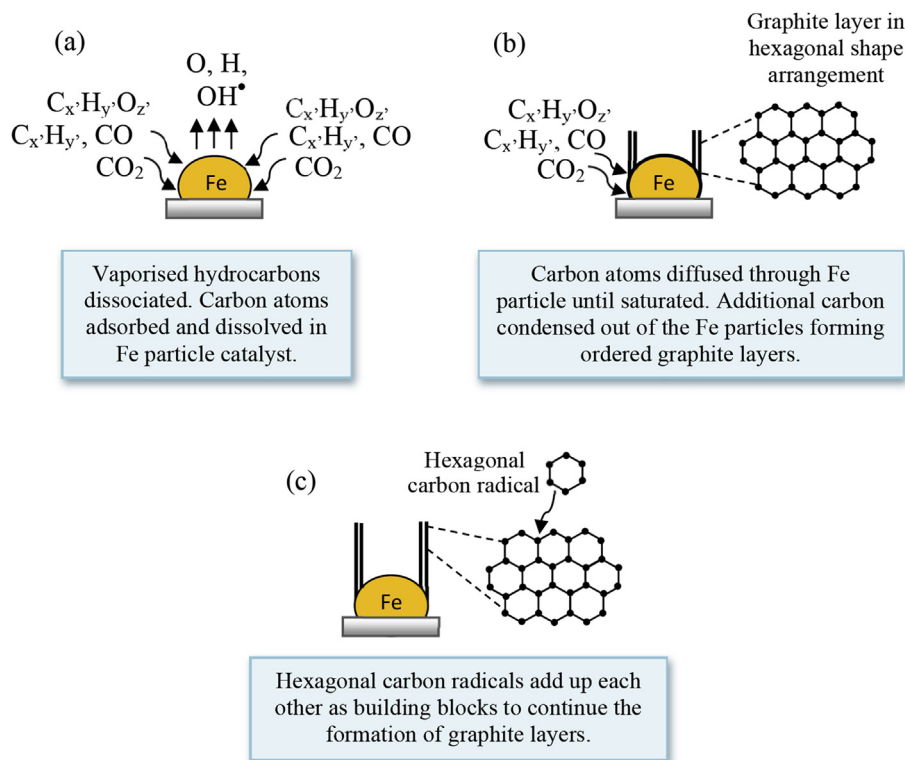
have a good graphitisation which leads to a high current conductivity.

In addition to FEE application, the potential mass production application of CNTs from gutter oil in supercapacitor device was also presented. Fig. 3 (d) shows the CV curve of the sample. The maximum specific capacitance ( $C_{sp}$ ) of the electrode was calculated using the following equation:

$$C_{sp} = 2I/(SR \times m) [30]$$

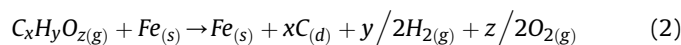
where  $I$  is the maximum measured current (A),  $SR$  is the scan rate (V/s), and  $m$  is the mass of the electrode.  $C_{sp}$  of the sample was calculated to be  $\sim 0.51 \text{ F/g}$ . The oxidation and reduction peaks were significant at the potential of  $\sim 0.20$  and  $\sim 0.18 \text{ V}$  suggesting the CNTs synthesised from gutter oil have a good electrochemical activity. This result paves the way to the development of alternative and potential mass production application of energy storage devices based on CNTs derived from gutter oil precursor.

A schematic diagram of the growth mechanism of quasi-aligned CNTs produced from gutter oil is shown in Fig. 4. The suggested



**Fig. 4.** Schematic diagram of the growth mechanism of the CNTs produced from gutter oil.

cracking pattern for gutter oil is similar to previous report [20]. The vaporised hydrocarbons would readily dissociate and carbon atoms are adsorbed and dissolved in the Fe particle catalyst (Fig. 4 (a)). The chemical reaction during these processes is shown in Eq. (2).



where  $xC_{(d)}$  is the carbon dissolved in catalyst.

The process continues with the diffusion of carbon atoms through Fe particles until saturation is achieved [20]. The additional carbon condensed out of the Fe particles (Fig. 4 (b)) forming ordered graphite layer on the surface of the catalyst particle in a hexagonal shape arrangement [45]. The growth process is also believed to be assisted by the aromatic hydrocarbon compound (ethylbenzene) contained in the gutter oil. The aromatic hydrocarbon consists of hexagonal ring that is an essential constituent of fullerene and tube-like structures gives an advantage to the growth of CNTs [9]. At a synthesis temperature of 800 °C, it was believed that most of the carbon, hydrogen and oxygen are detached, leaving the reactive hexagonal carbon radicals. The hexagonal carbon radical would only need to join together as building blocks to continue the formation of graphite layer [9] as shown in Fig. 4 (c).

#### 4. Conclusions

In summary, quasi-aligned CNTs with diameter between 30 and 50 nm, good crystallinity ( $I_D/I_G$  ratio of 0.66), and purity of 81% were obtained by pyrolysis of gutter oil. The measured FEE from these CNTs yield a high  $\beta$  value of 6158, low turn-on and threshold electric field values of 1.94 and 2.94 V  $\mu\text{m}^{-1}$ , respectively. The synthesised CNTs also open up potential mass production application in energy storage devices, such as supercapacitors. This study demonstrates that gutter oil, a low-cost and readily available resource, can be used as an inexpensive carbon source for the production of CNTs. This project not only describes a new application of gutter oil and FOG in the field of nanotechnology, but also offers a solution to the environmental pollution of ecosystems caused by the improper FOG disposal.

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