Effects of charcoal composition and oil droplet combustion on the polycyclic aromatic hydrocarbon content of smoke during the grilling process

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Abstract

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Introduction

250°C. those involving grilling and barbecuing, benzo [a] pyrene is defined as a marker for PAH content, and Processed chicken products are Thailand's most the maximum allowed level is 0.005 μ g g⁻¹ of wet exported meat product. In 2011, Thailand exported weight. In addition, the maximum combined level for approximately 415,585 tons of processed chicken benzo [a] pyrene, chrysene, benzo [a] anthracene, and worth 57,045 million Baht (Office of agricultural benzo [b] fluoranthene is 0.03 μ g g⁻¹ of wet weight. economics 2012). Yakitori, a charcoal-grilled meat Several studies

have demonstrated product, is one of the most important cookedcarcinogenic PAHs can be formed by the grilling and chicken products in the export market of the country. smoking of foods. The level of contamination by PAHs However, the charcoal-grilling process may cause during grilling depends on many factors, including the contamination of yakitori with carcinogenic incomplete combustion of charcoal (Farhadian et polycyclic aromatic hydrocarbons (PAHs). al. 2011), transformation of meat nutrients, such as PAHs are organic compounds which consist triglyceride and cholesterol, at high temperature, fat of at least two fused aromatic rings (Farhadian dripping onto hot charcoal (Farhadian et al. 2011; et al. 2011). Although more than 100 PAHs have Alomirah et al. 2011), and grilling time (Chen and

> Lin 1997). Charcoal is one of the main factors contributing to PAHs contamination in grilled meat. PAHs emissions from charcoal were detected in the smoke during the grilling of meat using charcoal as fuel (Dyremark et al. 1995) and these PAHs can deposit onto grilled foods (Chung et al. 2011). Olsson and Petersson (2003) reported that charcoal with a high carbon content released fewer aromatic compounds, including PAHs, during combustion. The composition of charcoal, especially the carbon content, depends on many factors, such as carbonization temperature,

in the atmosphere (Chen and Chen 2001). These priority 16 PAHs are naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[a]anthracene, chrysene, benzo [b] fluoranthene, benzo [k] fluoranthene, benzo [a] pyrene, dibenzo [a,h] anthracene, benzo [g,h,i] perylene, and indeno [1,2,3-c,d] pyrene. Moreover, according to EU Regulation No.835/2011 for smoked meat products and heat treated meat products, i.e.,

been characterized, the United States Environment

Protection Agency (U.S.EPA) identified 16 of them as

priority PAHs due to their carcinogenicity and presence



Combustion of charcoal and combustion of oil droplets may lead to food contamination with carcinogenic polycyclic aromatic hydrocarbons (PAHs) during the grilling process. This work aimed to determine the effect of charcoal composition and oil droplet combustion independently on 16 priority PAHs in the smoke produced during the grilling process. Charcoal prepared at 500°C produced 18% volatile matter which is composed of 5.8 µg PAHs per gram of dry charcoal. In contrast, charcoal produced at 750°C and 1,000°C presented 7-8% volatile matter and 0.57-0.69 µg PAHs per gram of dry charcoal. Furthermore, the effect of oil combustion on the PAH content in smoke was determined. The temperature of the heating source significantly affected the PAH content, which was 16.63 and 105 μ g PAHs per gram of oil at 250°C and 400°C, respectively. PAHs with higher molecular weights were formed at 400°C compared to

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that

wood type (Viegas *et al.* 2012), and carbonization method (Antal *et al.* 1996).

High carbonization temperatures lead to the formation of charcoal with high levels of carbon and volatile matter. Mangrove wood is widely used to produce charcoal in Thailand because it produces little ash and the charcoal has a high heating value (Ahmed *et al.* 2012). Therefore, in this work, the effect of carbonization temperature on the composition of mangrove charcoal, and on the PAH smoke content in smoke during the combustion, was studied. This knowledge is expected to reduce PAH content in grilled meat by indicating the proper charcoal preparation method.

Furthermore, melted fat droplets falling onto the charcoal can cause PAHs contamination of grilled food, as PAHs present in the smoke arising from the pyrolysis of fat droplets can directly deposit onto its surface (Farhadian et al. 2011). The amount of benzo[a]pyrene is directly proportional to the fat content during charcoal grilling (Doremire et al. 1979). However, to date, none of the studies has specifically analyzed the effect of fat combustion on the PAH content of the smoke produced. Therefore, in this work, the effect of different heating source temperatures, independent of the charcoal type, on the PAH smoke content has been studied by using an electrical hot plate as the heating source. Knowledge of the PAH content in smoke from the addition of oil at different temperatures is expected to indicate appropriate conditions for the grilling process, thereby reducing the PAH contamination of yakitori.

As charcoal grilling gives the unique aroma and color of yakitori, it is necessary to use charcoal as a heating source. Therefore, to understand each factor affecting the contamination of PAHs in yakitori products, this work aimed to determine the effect of charcoal composition and oil combustion independently on the smoke content of 16 PAHs during the grilling process. Firstly, the effect of charcoal composition on the PAH levels in the smoke during charcoal burning was studied. Mangrove charcoal with different compositions prepared at carbonization temperatures of 500°C, 750°C and 1,000°C were burned, and the smoke produced was collected and analyzed to determine the PAH content. Secondly, the effect of oil droplet combustion on the PAH content of the smoke was determined. Chicken oil was dropped onto an electrical heating source at 250°C and 400°C, and the smoke generated was collected and analyzed for PAH content. In all experiments, the smoke from charcoal burning and oil combustion was collected in an absorption tube from which the PAHs were extracted and then

analyzed using GC/MS.

Materials and Methods

Ten-year-old mangrove wood samples of 2 inch diameter were purchased from the local area in Khao Yeesarn (Samutsongkhram, Thailand). Chicken skin was obtained from B. Foods Product International Co. Ltd. (Lopburi, Thailand) and stored at -20°C prior to use. An ORBO-1500 PUF/XAD-2/PUF absorption tube and 16 mixed PAHs were purchased from Supelco (Bellefonte, USA) and Cambridge Isotope Laboratories (Massachusetts, USA). A Sep-Pak Florisil cartridge was obtained from Waters (Milford, USA). HPLC and AR grade chemical reagents were purchased from Merck (Darmstadt, Germany), Fisher Chemical (UK), Carlo Erba Reagents (Italy) and RCI LabScan (Bangkok, Thailand).

Determination of carbonization temperature on charcoal composition

To produce charcoal of different compositions, 100 g mangrove wood samples were placed in a ceramic box and then carbonized to charcoal in a furnace at 500°C, 750°C and 1,000°C. The temperature was increased at a rate of 20°C min⁻¹, then, once achieved, the desired temperature was maintained for 1 hour.

The composition of the charcoal obtained at 500°C, 750°C and 1,000°C were analyzed in terms of moisture, volatile matter, ash, and fixed carbon, according to the standard method outlined in ASTM D 3173-95. In addition, the yield of charcoal was calculated.

Determination of charcoal composition on PAH content of charcoal smoke

To determine the effect of charcoal composition on the PAH content of the smoke burned, 70 g of charcoal was burned in a furnace at 650°C at a heating rate at 20°C min⁻¹. During charcoal combustion, smoke was pumped from the furnace at 1.5 L min⁻¹ via a heat resistant rubber tube and collected in a PUF/XAD-2/PUF adsorption tube.

Determination of PAHs in smoke from chicken oil combustion at different heating source temperatures

During grilling, the combustion of oil droplets which fall onto the charcoal at different temperatures may generate different PAHs. Therefore, PAHs from the combustion of chicken oil droplets at different temperatures were determined. The melted oil from chicken skin was prepared by using an electrical hot plate heating at 60°C (IKA, model C-MAG HS 7,

PAHs	Retention time	Primary ion	Secondary ion
	(min)		
D8-Napthalene (Internal standard)	5.27	136	108, 137, 134
Naphthalene	5.28	128	129, 127, 51
Acenapthylene	6.85	152	153, 151, 76
Acenaphthene	7.02	153	154, 152, 76
Fluorene	7.53	166	165,167, 82
Phenanthrene	8.49	178	176, 17 9, 76
Anthracene	8.54	178	179, 176, 177
Fluoranthene	9.68	202	203, 200, 201
Pyrene	9.88	202	203, 200, 201
D10-Pyrene (Surrogate)	9.87	212	211, 100, 208
Benzo[a]anthracene	11.11	228	226, 229,114
Chrysene	11.11	228	226, 229, 227
Benzo[b]fluoranthene	12.05	252	253, 250, 126
Benzo[k]fluoranthene	12.06	252	253, 250, 1 26
Benzo[a]pyrene	12.44	252	253, 250, 1 26
Indeno[1,2,3-c,d]pyrene	13.82	276	277, 274, 138
Dibenzo[a,h]anthracene	13.86	278	279, 276, 139
Benzo[g,h,i]perylene	14.20	276	277, 138, 137

Table 1. Retention time, primary and secondary ions of 16 PAHs, plus internal and surrogate standard

Selangor, Malaysia). Heating source temperatures of 250±1°C and 400±1°C were used for the combustion of the dropping oil.

For the experimental setup of oil droplet combustion and smoke collection, one gram of chicken oil was dropped from a syringe onto a heating block (Tecator, model 1002, Hoganas, Sweden), one droplet at a time, at 250°C or 400°C. Smoke generated from the chicken oil combustion was passed through a PUF/XAD-2/PUF absorption tube by air pump (Resun, model AP – 40, China) at 2.5 L min⁻¹.

Determination of PAHs using GC/MS

The PAHs were extracted from the adsorption tube according to the method outlined in TO-13A by the United States Environment Protective Agency, 1999. Briefly, PAHs were extracted from the PUF portion of the adsorption tube with 350 mL of 10% diethyl ether in hexane, and from the XAD-2 resin portion with 350 mL of methylene chloride, for 18 hours using soxhlet extraction. After extraction, the solvent was filtered through 10 g of anhydrous sodium sulfate to remove water and evaporated under reduced pressure. The residue was dissolved in 1.00 mL of hexane, filtered through a 0.2 µm membrane filter, and stored in a vial.

A 7890A GC System (Agilent Technologies, Canada) fitted with a DB-5MS column (30 m length x 0.25 mm i.d., Agilent Technologies, USA) with 0.25 μ m of film thickness coupled to a 5975C inert XL MSD (Agilent Technologies, USA) with an auto sampler was used to determine the PAHs. The injection volume was 2 μ L. The oven temperature program was kept at 55°C for 1 min, increased to 320°C at 25°C min⁻¹, and then maintained at 320°C for 3 min. Helium was used as the carrier gas. The MS transfer line was kept at 280°C (Lynam and Smith 2008). Electron ionization mass spectra were recorded at 70 eV, and the mass range detected was from 45 to 450 atomic mass units. The analysis was set to SIM mode, and the PAH data was considered valid when the Q value was more than 80 (Chen and Chen 2003).

Sixteen carcinogenic PAHs were identified by gas chromatography and mass spectrometry. Table 1 shows the retention time, primary and secondary ions of 16 PAHs, plus internal and surrogate standard.

Results and Discussion

Carbonization temperature on charcoal composition

Different charcoal components, especially the volatile matter, may form different PAHs during grilling (Olsson and Petersson 2003), and these PAHs may directly deposit onto the surface of food (Chen and Lin 1997). Therefore, in this study, charcoals with different compositions were produced at different carbonization temperatures. Then, the obtained charcoal was burned in a furnace at 650°C to determine the PAH content of the smoke generated.

Mangrove charcoal was produced at carbonization temperatures of 500°C, 750°C and 1,000°C. The charcoal compositions were analyzed in terms of moisture content, volatile matter, ash,

Composition	Carbonization temperature (°C)			
(%)	500	750	1,000	
Moisture	0.09±0.02	0.56±0.02	0.74±0.06	
Volatile matter	18.45±0.14	7.91±0.32	7.73±0.36	
Ash	4.39±0.00	5.12±0.33	6.53±0.28	
Fixed carbon	77.15±0.13	86.95±0.68	85.72±0.34	
Yield	28.42±0.09	22.07±2.48	21.22±0.33	

Table 2. Moisture content, volatile matter, ash, and fixed carbon content of mangrove charcoal carbonized at 500°C, 750°C, and 1,000°C

fixed carbon, and yield, as shown in Table 2. The carbonization temperature significantly affected the charcoal composition and yield. Charcoal produced at 500°C presented the highest volatile matter content (18%) and yield (28%), but had the lowest fixed carbon content (77%). In contrast, charcoal produced at 750°C and 1,000°C presented a low volatile matter content (7%) and yield (21-22%), but had a higher fixed carbon content (more than 80%). However, no significant differences in the composition of charcoal produced at 750°C and 1,000°C were observed. These results indicate that higher carbonization temperature creates more pure charcoal due to a lower volatile matter content and a lower yield.

These results show the same trend as research (Demirbas 1999) which reported that different charcoal compositions were obtained from hazelnut shell carbonized at different temperatures. At a high carbonization temperature (777°C), the charcoal derived from hazelnut shell was purer and had a low volatile matter content, a high fixed carbon content, and a high heating value.

Normally, pyrolysis of wood biomass, i.e., cellulose and hemicelluloses, is achieved at temperatures of around 400°C. Tar, a residue formed in charcoal production, is in the form of a liquid at room temperature and becomes volatile at high temperature, typically above 850°C (Antal *et al.* 1996). Thus, carbonization temperatures of 750°C and 1,000°C lead to a decrease in the volatile matter content of the charcoal.

PAH content in the smoke of charcoal with different compositions

As volatile matter in charcoal may form PAHs at grilling temperature, these PAHs may arise from charcoal smoke and directly deposit onto the surface of food. Therefore, charcoal with different compositions especially volatile matter, was burned to determine the PAH content generated in smoke.

Table 3 shows the concentrations of PAHs in the smoke generated by the burning of charcoal

with different carbonization temperature. Charcoal produced at 500°C, containing 18% volatile matter, presented the highest concentration of the 16 PAHs at around 5.8 μ g/g of dry charcoal. In contrast, charcoal produced at 750°C and 1,000°C, containing 8% and 7% volatile matter, respectively, presented PAH contents of just 0.57 and 0.69 μ g g⁻¹ of dry charcoal. This observation is supported by Fjellerup *et al.* (2005), who reported that when tar is heated, PAHs, including naphthalene, and phenanthrene, are formed.

According to EU regulation No. 835/2011, the maximum level of benzo [a] pyrene in heat treated meat products should not exceed 0.005 μ g g⁻¹ of wet weight, and the total content of benzo [a] pyrene, chrysene, benzo [a] anthracene, and benzo [b] fluoranthene should not be more than 0.03 μ g g⁻¹ of wet weight. Since PAHs are lipophilic, they adhere to food during charcoal grilling. Table 3 shows that the levels of benzo[a]pyrene and the 4 PAHs above in the smoke from charcoal carbonized at 500°C are 0.02 and 0.28 μ g g⁻¹ of dry charcoal, respectively. However, none of these PAHs were detected in the smoke from the charcoal carbonized at 750°C and 1,000°C.

Therefore, it is possible that food grilled using charcoal carbonized at low temperature presents a high risk of PAH contamination. However, this aim of this study was not to determine the amount of PAHs that adhere to charcoal-grilled food. Consequently, the contamination of charcoal-grilled food by PAHs will be investigated in later research.

PAHs in smoke from the combustion of chicken oil droplets at different temperatures

During grilling, the combustion of oil droplets on charcoal at different temperatures may generate different PAHs. Therefore, PAHs from the combustion of chicken oil droplets at different heating source temperatures was determined using an electrical hot plate as the heating source in order to eliminate the effect of charcoal combustion.

PAHs	PAHs concentration (µg/g dry charcoal)			
	Carbonization temperature (°C)			
	500	750	1,000	
Naphthalene	2.19±1.88	0.46±0.45	0.53±0.74	
Acenaphthylene	0.19±0.26	N.D.	N.D.	
Acenaphthene	N.D.	N.D.	N.D.	
Fluorene	N.D.	N.D.	N.D.	
Phenanthene	1.22±1.03	0.05±0.04	0.02±0.03	
Anthracene	0.35±0.25	0.003±0.004	0.02±0.03	
Fluoranthene	0.67±0.94	N.D.	0.06±0.07	
Pyrene	0.89±1.02	0.05±0.01	0.06±0.07	
Benzo[a]anthracene*	0.13±0.16	N.D.	N.D.	
Chrysene*	0.10±0.12	N.D.	N.D.	
Benzo[b]fluoranthene*	0.03±0.04	N.D.	N.D.	
Benzo[k]fluoranthene	N.D.	N.D.	N.D.	
Benzo[a]pyrene*	0.02±0.03	N.D.	N.D.	
Indeno[1,2,3-c,d]pyrene	0.01±0.01	N.D.	N.D.	
Dibenzo[a,h]anthracene	N.D.	N.D.	N.D.	
Benzo[g,h,i]perylene	0.005±0.01	N.D.	N.D.	
16 PAHs	5.79	0.57	0.69	
4 PAHs*	0.28	N.D.	N.D.	

Table 3. Concentrations of PAHs in smoke from charcoal produced at different carbonization temperatures, burning at 650°C

Table 4 shows the PAH content of smoke generated by dropping oil onto the heating source. Research has shown that oil combustion generates PAHs (Chen and Chen 2001; Chen and Lin 1997), but the mechanism of formation was not clear. However, research (Chen and Chen 2001) proposed that benzene may be a precursor for PAH formation, and that molecules containing double bonds can form benzene ring-containing compounds via the Diels-Alder reaction. Moreover, polyunsaturated fatty acids can undergo internal cyclisation and polymerization to form cyclic monomers or dimers. The chicken oil in this study consists of double bond-containing molecules, such as fatty acids and polyunsaturated acids, PAHs may form through these reactions.

The heating source temperature significantly affected the PAH content of the smoke generated. When oil is dropped onto a heating source, thermal cracking, or pyrolysis, of the oil occurs, causing PAH formation (Maher and Bressler 2007). In our experiments, the PAH content of the smoke was 16.63 and 105 μ g g⁻¹ of oil at heating source temperatures of 250°C and 400°C, respectively. Moreover, oil pyrolysed at 250°C presented more types of PAHs than that pyrolysed at 40°C. Therefore, high temperature presents more PAH types, and at higher levels. This may be explained by the increase of reaction rates at higher temperatures. Temperature is known to have a significant effect on the rate of

oil pyrolysis. Hampikyan and Colak (2010) reported that the temperature of smoke plays an important role in PAH content, and the amount of PAHs in smoke formed during pyrolysis increases linearly with smoke temperatures between 400°C -1,000°C.

In addition, it was observed that PAHs with high molecular weights were produced at high temperature. It is possible that low molecular weight PAHs are converted to high molecular weight PAHs when the temperature is increased. This theory is supported by Houessou *et al.* (2007) who reported the transformation of PAHs from low to high molecular weight in roasting coffee.

As shown in Table 4, no benzo[a]pyrene was found in the smoke from oil droplets pyrolysed at 250°C, but the other 4 regulated PAHs mentioned previously were present at a level of 0.24 μ g g⁻¹ of oil. At 400°C the smoke produced by oil combustion contained benzo[a]pyrene at a level of 0.10 μ g g⁻¹ of oil, and the other 4 regulated PAHs were present at a level of 0.39 μ g g⁻¹ of oil. Assuming that all PAHs generated from oil combustion contaminate the grilled food, the maximum level defined in EU regulation No 835/2011 will be exceeded.

Conclusions

Charcoal-grilling gives the unique aroma and color of yakitori. However, charcoal combustion and

PAHs	PAHs concentration µg/ g oil (ppm)		
	250°C	400°C	
Naphthalene	14.76±9.39	66.12±35.88	
Acenaphthylene	N.D.	N.D.	
Acenaphthene	N.D.	N.D.	
Fluorene	1.12±0.01	11.19±1.50	
Phenanthene	0.52±0.08	14.45±10.34	
Anthracene	N.D.	11.17±15.52	
Fluoranthene	N.D.	N.D.	
Pyrene	N.D.	1.61±1.23	
Benzo[a]anthracene*	0.24±0.33	N.D.	
Chrysene*	N.D.	0.21±0.14	
Benzo[b]fluoranthene*	N.D.	0.08±0.03	
Benzo[k]fluoranthene	N.D.	N.D.	
Benzo[a]pyrene*	N.D.	0.10±0.12	
Indeno[1,2,3-c,d]pyrene	N.D.	0.02±0.0	
Dibenzo[a,h]anthracene	N.D.	0.03±0.05	
Benzo[g,h,i]perylene	N.D.	N.D.	
16 PAHs	16.63	105.00	
4 PAHs*	0.24	0.39	

Table 4. PAH concentration (µg/g of oil) in smoke from combustion of chicken oil droplets at 250°C and 400°C

oil droplet combustion can cause PAH contamination of grilled food. The understanding of each factor affecting the PAH contamination of yakitori products is expected to reduce the PAH contamination in Yakitori and other grilled meat. This knowledge can be used to inform producers and consumers about the proper conditions needed for charcoal-grilling. The results of our study indicated that volatile matter in charcoal affected the PAH levels in the smoke produced during charcoal burning. Mangrove charcoal carbonized at 500°C contained 18% volatile matter and presented a PAH level of 5.8 μ g g⁻¹ of dry charcoal. In contrast, charcoal produced at 750°C and 1,000°C contained 7-8% volatile matter and presented PAHs at a level of just 0.57-0.69 µg g⁻¹ of dry charcoal. In addition, the temperature of the heating source used to pyrolyse oil droplets affected the PAH level in the smoke produced. The PAH content was 16.63 and 105 µg g⁻¹ of oil at 250°C and 400°C, respectively. Moreover, oil dropped onto a heating source at a temperature of 400°C produced more types of PAH with higher molecular weights than those formed at 250°C.

Therefore, it is possible that food grilled using charcoal carbonized at low temperatures has a high risk of PAH contamination. Consequently, food contamination by PAHs generated in charcoal smoke will be further studied. In addition, the combined effects of oil combustion and charcoal preparation on the levels of PAHs deposited onto chicken meat will be taken into account in our further study.

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