



Emission of polycyclic aromatic hydrocarbons from the pyrolysis of liquid crystal wastes

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ABSTRACT

A liquid crystal display can be described as a panel consisting of two plates of glass with liquid crystals in the space between. Generally, the liquid crystal wastes are extracted and separated into various fractions. Some recyclable materials, i.e., metals, glass, plastics, etc., are recycled, but the liquid crystals are incinerated. The emission factors for 16 U.S. EPA priority polycyclic aromatic hydrocarbons (PAHs) from the combustion of liquid crystal are approximately 390 and 1520 times higher than that of waste terephthalic acid and biological sludge combustion, respectively. In this study, we determined the emission of PAHs from the liquid crystals pyrolysis. We also investigated the fragments and gas compositions using on-line thermogravimetry–mass spectrometry (TG–MS). A temperature series of 14 fragments was analyzed in nitrogen, and was found to include m/z : 30, 32, 42, 44, 55, 57, 67, 81, 95, 109, 128, 166, 178, and 202. The fragments at m/z 32 represents formaldehyde and the fragment at m/z 44 is carbon dioxide. The fragments at m/z 55, 57, 67, 81, 95, and 109 represent hydrocarbon components, all of which may be liquid crystal by products. The TG–MS as analyzed above can offer a better understanding of the mechanisms of byproduct formation in liquid crystal waste pyrolysis.

Experimentally, not detected (n.d.) –5.98 and n.d. –20.2 $\mu\text{g/g}$ of 16 PAHs, in the particulate and gas phases, respectively, are determined from the emission of liquid crystal waste pyrolysis. The PAH profiles showed a predominance of naphthalene (42.6%) and phenanthrene (13.5%). The total PAH emissions for the 16 species were 7.75 and 44.05 $\mu\text{g/g}$ in the particulate and gas phases, respectively, significantly lower than the values associated with liquid crystal combustion. From the viewpoint of PAH emission control, our results suggest that the pyrolysis is a better option for the disposal of liquid crystal wastes than that of combustion.

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1. Introduction

Liquid crystal displays (LCDs) are widely used in televisions, mobile phones, portable computers, PC monitors, pocket calculators, etc. Due to the large quantity of LCDs in use, the volume of liquid crystal waste is increasing very fast, causing growing concern about its economic and environmental impact. In general, liquid crystal wastes mainly contain waste plastics, glass and liquid crystals. Most of the liquid crystal wastes including those from portable computers, PC monitors and LCD TVs are recycled under electronic wastes collecting schemes. They are disassembled and separated into various components. Certain valuable materials, i.e., metals, glass, plastics, etc., are recycled, but the liquid crystals are incinerated.

It is well known that combustion technology offers many advantages, including maximum volume reduction, permanent disposal and detoxification. However, certain undesirable air pollutants, such as PAHs, are generally produced in the combustion process. PAHs are of special interest due to their toxicity, carcinogenicity and ubiquitous presence in the environment [1]. In particular, some of the pyrogenic PAHs are on the U.S. EPA list of priority pollutants [2]. In a previous study, we found that the liquid crystal combustion emits n.d. –25.19 and n.d. –31.48 $\mu\text{g/g}$ for 16 U.S. EPA priority PAHs in the particulate and gas phases, respectively [3]. The emission factors are approximately 390 and 1520 times higher, respectively, than those of waste terephthalic acid and biological sludge combustion. From the point of view of air pollution control, it is necessary to develop an alternative method for the disposal of liquid crystal wastes. In addition, since certain PAHs are potentially mutagenic and carcinogenic, their identification and minimization are essential during the thermal process.

Pyrolysis has been proven to be an effective alternative for resource recovery. The pyrolysis of wastes has been investigated in the context of the disposal of waste printed circuit boards [4],

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scrap tires [5], and waste plastics [6], among others. In a previous study, the feasibility of liquid crystal waste pyrolysis was investigated [7]. The liquid crystals can be effectively decomposed at 413–596 K. The activation energy, pre-exponential constant and reaction order of the pyrolysis of liquid crystals are 17.0 kcal mol⁻¹, 10^{8.71} min⁻¹ and 1.15, respectively. The global rate equation for the pyrolysis of the liquid crystals can be expressed as $dX/dt = 10^{8.71} [\exp(-17.0/(8.314 \times 10^{-3}T))](1-X)^{1.15}$ (X denotes the reaction conversion). Thus, it is apparent that the pyrolysis may be an alternative disposal option for liquid crystal wastes. However, to the best of our knowledge, PAH emissions from the pyrolysis of liquid crystal wastes have never been investigated.

Simultaneous TG–MS is an excellent tool to investigate the mechanisms of thermal decomposition. It provides direct real-time analysis information about fragments or gas compositions in the context of thermal decomposition. TG–MS can be used to obtain a deeper understanding of the pyrolysis process. For example, Perng used TG–MS to investigate the mechanism of poly(phenylene sulfide) pyrolysis [8]. Várhegyi et al. analyzed the gas emission characteristics of biomass charcoal pyrolysis by the TG–MS technique [9]. Fushime et al. also used TG–MS to measure gas emission profiles in steam gasification of biomass [10]. TG–MS can also be used to study the properties of a catalyst for post-combustion soot scenarios [11]. The objective of this paper is to determine the emission of PAHs from the pyrolysis of liquid crystals. The fragments and gas compositions are also determined by TG–MS spectroscopy. This study provides useful information to the public regarding PAH emissions from liquid crystal waste pyrolysis.

2. Experimental

The sample of liquid crystals used in this study comes from commercial-grade material that is used in the fabrication of 14 in. portable computers and 17 in. LCDs in Taiwan. Note that typical liquid crystals contain 77.1 wt.% of C, 8.4% of H and 8.5 wt.% of N and O (balanced) [7].

The liquid crystal pyrolysis experiments were conducted in a fixed-bed reactor (SS 316 tube, ID = 2.54 cm and $L = 45$ cm). About 3.0 g of liquid crystals were heated from room temperature to 673 K under high-purity nitrogen at a flow rate of 10 mL min⁻¹ (gas hourly space velocity (GHSV) of approximately 200 h⁻¹). A water-cooled heat exchanger was used to refrigerate the gas products. Glass fiber filters (MF-Millipore, 0.45 μ m) and XAD-2 resins (SUPELCO) were used to collect the particulate and gas phase PAHs, respectively, that were sequentially connected to the heat exchanger.

Prior the pyrolysis experiments, the glass fiber filters and XAD-2 resins were precleaned by Soxhlet extraction for 24 h in methylene chloride. After completion of the pyrolysis run, they were removed and placed in separate glass bottles with Teflon-lined caps and stored at 277 K. The filters and resins were placed in the precleaned Soxhlet apparatus again and extracted for 24 h in methylene chloride. The extracted liquids were subsequently concentrated to a volume of 1 mL using high-purity nitrogen, and transferred to a Teflon-sealed screw-cap vial for storage and analysis. To ensure the purity of the XAD-2 resins and glass fiber filters, blanks of XAD-2 resins and glass fiber filters were also extracted and analyzed. No PAHs were detected in the plain XAD-2 and glass fiber filter extracts. For the solution blank test, 250 mL methylene chloride was concentrated to 1 mL. The solution was injected into the gas chromatography/mass spectroscopy (GC/MS) apparatus to assure that there were no detectable PAHs in the solvent used. In addition, PAHs were also not detected from sampling the reactor exhaust without any liquid crystals present (pyrolysis blanks).

We also tested the recovery of PAHs from the XAD-2 resins and glass fiber filters. Standard amounts of standard PAH solutions were

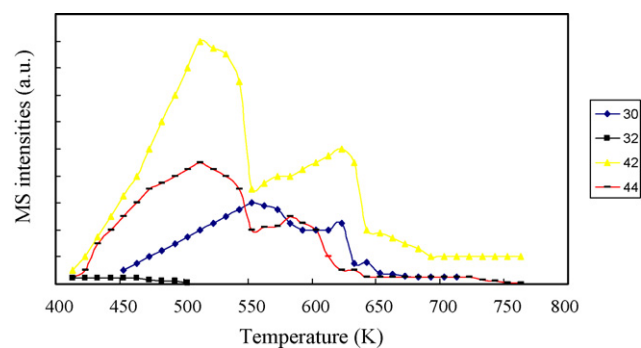


Fig. 1. Mass spectroscopy intensity curves of $m/z = 30, 32, 42$ and 44 from liquid crystal pyrolysis.

placed in the XAD-2 resins and glass fiber filters, respectively, which were then sequentially subjected to Soxhlet extraction, concentration to 1 mL and analysis for the PAH levels. The recoveries (in duplicate repetitions) of the PAHs for the XAD-2 resins and glass fiber filters were 81.2–95.1% and 85.1–93.5%, respectively. A duplicate analysis was conducted to assess the reproducibility of the PAH analysis.

The concentrations of 16 PAH compounds were measured by GC/MS using a SHIMADZU GC QP 2010 equipped with a SHIMADZU MS QP 2010 mass selective detector. The GC was fitted with a DB-5 (30 m \times 0.25 mm) column. The temperature program was as follows: injection port, 563 K; transfer line, 523 K; initial temperature, 343 K; initial hold, 1 min; ramp rate, 15 K min⁻¹; final temperature, 553 K; final hold, 15 min. The PAHs were confirmed and quantified using the retention times and calibration curves from the standard PAH solutions (Dr. Ehrenstorfer GmbH PAH-Mix 25). The analysis was performed using 1 μ L injections and helium gas was used as the carrier gas at a flow rate of 1.66 mL/min. Mass spectroscopy was performed in the selected ion monitoring (SIM) mode at an electron energy of 70 eV.

The TG–MS experiments were performed simultaneously using a TG (STA 409 CD, Netzsch Instruments, Inc.) and a quadrupole mass spectrometer (QMA 400, Balzers Instruments, Inc.) system. A Skimmer Coupling System (Netzsch Instruments, Inc.) was used to combine these two instruments. About 3.0 mg of liquid crystals were decomposed with TG, and the fragments and gas products were then introduced to the mass spectrometer to obtain evolution curves. The sample was heated to 773 K at a heating rate of 8 K min⁻¹ in 100 mL min⁻¹ high-purity nitrogen.

3. Results and discussion

The following m/z ratios were obtained from TG–MS from the decomposition mechanisms of liquid crystal pyrolysis: 30, 32, 42, 44, 55, 57, 67, 81, 95, 109, 128, 166, 178 and 202. The temperature series of selected MS fragments are shown in Figs. 1–3. Since the evolution of gases begins at about 413 K and since concentrations continue to increase gradually with increasing temperatures until 623 K, one may hypothesize that the pyrolysis reaction starts at 413 K, which is consistent with the previously reported TG results [7]. In Fig. 1, the intensities increased at low temperatures, showed two peaks at around 513 and 623 K and decreased at temperatures above 633 K. $m/z = 32$ represents formaldehyde, which is consistent with the argument proposed by Lüftl et al. [12]. The pyrolysis of an oxygen-containing compound can produce formaldehyde and similar fragments. In addition to hydrocarbon fragments, the temperature series of CO₂ ($m/z = 44$) is also shown in Fig. 1. As expected, since liquid crystals contain 6.0% of oxygen, CO₂ is also observed in the TG–MS analysis. However, because the TG–MS experiments are conducted under a nitrogen

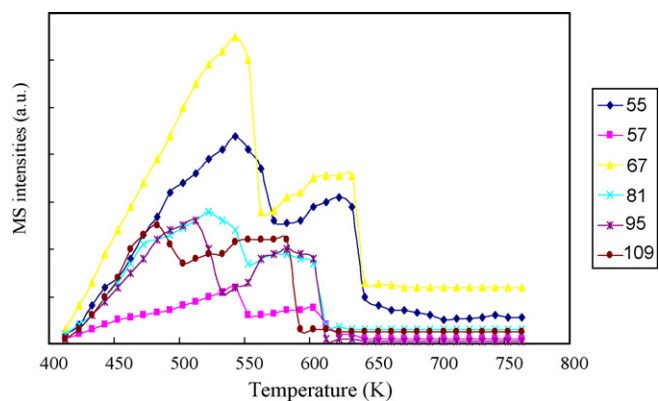


Fig. 2. Mass spectroscopy intensity curves of $m/z=55, 57, 67, 81, 95$ and 109 from liquid crystal pyrolysis.

atmosphere, we were not able to track the m/z ratio of CO or C_2H_4 .

The m/z ratios plotted in Fig. 2 include: $m/z=57$ (C_4H_9^+), which may represent fragments of alkanes; 55 (C_4H_7^+), which may be fragments of alkenes; 67 (C_5H_7^+), 81 (C_6H_9^+), 95 ($\text{C}_7\text{H}_{11}^+$), and 109 ($\text{C}_8\text{H}_{13}^+$), which may be fragments of dienes. It is clear that there are double peaks for the fragment intensities spanning the whole temperature domain. These fragments may be produced from C–C or C=C bond cleavage of the long carbon chain of liquid crystals, depending on the numbers and locations of the C=C double bonds of the original liquid crystals. Since the main components of the liquid crystals are biphenyl, azomethine, pyrimidine or difluorophenylene, it is reasonable to expect that naphthalene, fluorene, anthracene and fluoranthene may be observed in the TG–MS analysis (in Fig. 3). These compounds may be expected as byproducts of liquid crystal pyrolysis. In addition, it is worth noting that NO and NO_2 were not found by TG–MS spectroscopy. This is consistent with the results of our previous study of on-line FTIR spectra [3].

The TG–MS information may be helpful in understanding the decomposition mechanisms of liquid crystals and the potential byproducts. However, the concentrations and emission factors cannot be determined by TG–MS analysis. Thus, the emission of PAHs from liquid crystal pyrolysis was also conducted on a fixed-bed reactor to collect PAHs samples.

The pyrolysis process of liquid crystals is very complex since the reaction takes place in both the liquid and gas phases, as well as at the interface between the two. The pyrolysis of liquid crystals is combined with evaporation, gas diffusion, and mass and energy transfer. The evaporation and gas diffusion of liquid crystals occur in the early stages of the pyrolysis process. The liquid crystals may be partially cracked by chain scission into smaller and unstable

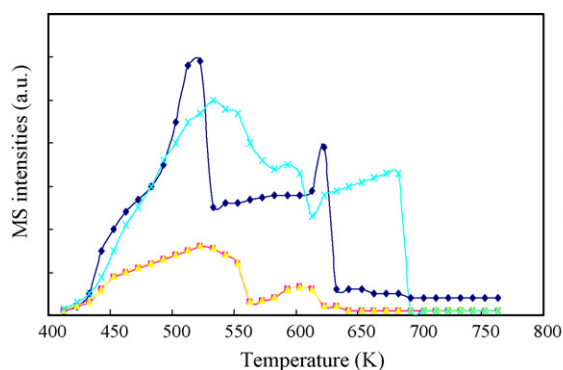


Fig. 3. Mass spectroscopy intensity curves of $m/z=128, 166, 178$ and 202 from liquid crystal pyrolysis.

fragments at higher temperatures. These fragments might mainly include highly reactive free radicals with very short lifetimes, which form hydrocarbons and CO_2 or that recombine to form more stable PAHs. These PAHs may exist in the particulate or gas phases depending on the reaction temperature and their molecular weights. The emission factors of the PAHs are expressed on the basis of the mass of PAHs emitted from each gram of liquid crystals in the pyrolysis. As shown in Table 1, the emission factors for 16 PAHs in the particulate and gas phases are n.d. –5.98 and n.d. –20.2 $\mu\text{g/g}$, respectively. The total PAH emissions for the sum of 16 species are 17.46 and 44.05 $\mu\text{g/g}$ in the particulate and gas phases, respectively, values that are significantly lower than those associated with liquid crystal combustion [3].

The partitions between the particulate and gas phases were about 72% and 28%, respectively. These results are consistent with the boiling points and van der Waals interactions between PAHs and particulate surfaces, which seem to increase with heavier molecular weights, similar to in the process associates with liquid crystal combustion, but quite different from the results of polystyrene combustion reported by Durlak et al. [13]. The total PAHs emitted from polystyrene combustion are 23.18 (65%) and 12.52 mg/g (35%) in the particulate and gas phases, respectively. On the other hand, as in biomass combustion [14] and liquid crystal combustion [7], naphthalene is observed to constitute a major share of the total PAHs. This is due to the fact that naphthalene has the lowest boiling point and the highest stability of the 16 PAHs. Jenkins et al. indicated that acidic vapors may cause depolymerization of XAD-2 resins, producing aromatic byproducts that obscure the analysis of naphthalene [15]. However, since naphthalene was not detected in the blank analysis of XAD-2, one may eliminate the possibility of the naphthalene contamination from XAD-2 in our PAH analysis.

It is known that the higher molecular weight, the higher the expected carcinogenic potency. The carcinogenic potency of a given PAH compound can be expressed in terms of its benz[*a*]pyrene (BaP) equivalent concentration (BaP_{eq}). The toxic equivalent factors (TEF) for a given PAH compound have been reported by Nisbet and LaGoy [16]. The total BaP_{eq} content for PAHs in the particulate and gas phases are 3.58 and 2.54 $\mu\text{g/g}$, respectively, much lower than those of liquid crystal combustion. From the viewpoint of control of PAH emissions, these results suggest that pyrolysis is a better alternative than combustion for the disposal of liquid crystal wastes.

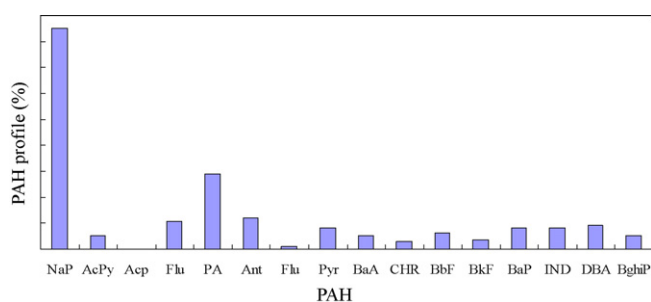
The PAH content also can be classified according to molecular weights: low molecular weight (LM-PAHs: containing 2–3-ring PAHs), middle molecular weight (MM-PAHs: containing 4-ring PAHs), and high molecular weight (HM-PAHs: containing 5–6-ring PAHs). Table 1 shows the trend of LM-PAHs > HM-PAHs > MM-PAHs in the pyrolysis of liquid crystals. Clearly, LM-PAHs were the dominant species in the pyrolysis of liquid crystals. Generally, only 2–3-ring PAHs are present in the gas phase, while heavier multi-ring PAHs (such as benzo[*a*]pyrene (BaP) and other compounds with 5 and 6 rings) are adsorbed onto particulate matter [17]. Durlak et al. [13] and Yang et al. [18] also indicated that the HM-PAHs appear primarily in the form of particulate state. Thus, since relatively large amounts of MM-PAHs and HM-PAHs were found in the particulate phase, our results indicate the need for good control of fine particulate emissions from the pyrolysis of liquid crystals.

To understand individual PAH emissions, it is important to assess the PAH emission profiles and characteristic ratios for the source apportionment profile. The individual PAH emission factors were normalized by the sum of 16 the PAHs to obtain PAH emission profiles. As shown in Fig. 4, the representative PAH profiles can be a useful source signature in PAH emission source apportionment analysis. The PAH profile shows significant proportions of naphthalene (42.6%) and phenanthrene (13.5%). Many studies have proposed that the ratios between PAH compounds can be used for source identification [18–20]. The calculated values for the most

Table 1

The emission factors for 16 U.S. EPA priority PAHs from the liquid crystal pyrolysis.

PAH compound	In gas phase ($\mu\text{g/g}$)	In particulate phase ($\mu\text{g/g}$)	Total ($\mu\text{g/g}$)	Detection limit ($\mu\text{g/g}$)
Naphthalene	20.2	5.98	26.2	0.00132
Acenaphthylene	1.64	n.d.	1.64	0.00079
Acenaphthene	n.d.	n.d.	n.d.	0.00141
Fluorene	2.94	0.32	3.26	0.00173
Phenanthrene	7.78	1.18	8.96	0.00169
Anthracene	2.78	0.92	3.70	0.00190
Fluoranthene	n.d.	0.32	0.32	0.00149
Pyrene	2.08	0.42	2.50	0.00141
Benz[a]anthracene	1.06	0.57	1.63	0.00185
Chrysene	0.88	n.d.	0.88	0.00378
Benz[b]fluoranthene	0.88	1.06	1.94	0.00182
Benz[k]fluoranthene	0.42	0.68	1.10	0.00303
Benz[a]pyrene	1.08	1.42	2.50	0.00988
Indeo[1,2,3,-cd]pyrene	0.71	1.77	2.48	0.00356
Dibenz[a,h]anthracene	1.08	1.72	2.80	0.00970
Benzo[ghi]perylene	0.52	1.10	1.62	0.00298
Total-PAHs	44.05	17.46	61.51	
LM-PAHs	35.34	8.40	43.74	
MM-PAHs	4.02	1.31	5.33	
HM-PAHs	4.69	7.75	12.44	
Total BaP _{eq}	2.54	3.58	6.12	

**Fig. 4.** PAH source profiles normalized by the sum of 16 PAHs.**Table 2**

Comparison of PAH characteristic ratios.

PAH ratios	Liquid crystal pyrolysis	Liquid crystal combustion [3]	Joss paper burning [18]
Pyr/BaP	1.00	0.50	1.2
BaP/BghiP	1.54	0.86	1.2
BghiP/IND	0.65	1.40	0.60
BaA/BaP	0.65	1.33	0.40

often used ratios of PAHs for liquid crystal pyrolysis, combustion, and joss paper combustion are listed in Table 2. The BghiP/IND ratio for the liquid crystal pyrolysis emission is closer to that of joss paper burning emissions. In addition, the other PAH ratios are quite different across emission sources. Furthermore, there is a variance even among the same feedstock, i.e., liquid crystal samples, subjected to different thermal processes, such as pyrolysis and combustion. This is because the PAH emissions are not only closely related to the feedstock, but also to with the thermal conditions and the type of appliance used. The results suggested that when the ratios between PAH compounds are used for source identification, in addition to the feedstock, the disposal conditions and the type of appliance should be noted.

4. Conclusions

We determined the level of PAH emissions from the pyrolysis of liquid crystals. In particular, the fragments and gas compositions were investigated by on-line TG–MS. The temperature series of 14 fragments were analyzed in nitrogen, and were shown to include

m/z : 30, 32, 42, 44, 55, 57, 67, 81, 95, 109, 128, 166, 178, and 202. The fragment at m/z 32 represents formaldehyde, and the fragment at m/z 44 is carbon dioxide. The fragments at m/z 55, 57, 67, 81, 95, and 109 represent hydrocarbon components, all of which may be fragments of liquid crystals. The TG–MS analysis above can offer a better understanding of the mechanisms of byproduct formation in liquid crystal waste pyrolysis.

The emission factors for 16 PAHs for liquid crystal waste pyrolysis are n.d. –5.98 and n.d. –20.2 $\mu\text{g/g}$ in the particulate and gas phases, respectively. The PAH profiles showed a dominance of naphthalene (42.6%) and phenanthrene (13.5%). The total PAH emissions for the sum of 16 species are 7.75 and 44.05 $\mu\text{g/g}$ in the particulate and gas phases, respectively, values that are significantly lower than those associated with liquid crystal combustion. The total BaP_{eq} content for PAHs is 6.12 $\mu\text{g/g}$. This value is also much lower than that for liquid crystal combustion. From the viewpoint of PAH emission control, these results suggest that pyrolysis is a better alternative for the disposal of liquid crystal wastes than combustion. In addition, since a relatively large quantity of heavier multi-ring PAHs are adsorbed onto particulate matter, the emission control strategies should emphasize the collection of fine particulates in the pyrolysis of liquid crystal wastes. Furthermore, while the ratios between PAH compounds can be used for source identification, the results suggested that in addition to the feedstock, the disposal conditions and the type of appliance should be noted.

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