



Correlation between the Concentration of Aromatic Hydrocarbons and BaP from Coke Oven Fugitive Emissions in Shanxi, China

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ABSTRACT

Most aromatic hydrocarbons are considered to be carcinogenic. Benzo[a]pyrene (BaP) is known to be a strong carcinogen which is typically found in coke plant emissions. When the waste gas finally exits through an exhaust funnel, some of this is emitted directly from leaks in the coke oven or flue. Authorities in Shanxi attempted to develop a real-time monitoring system for BaP in coke oven fugitive emissions. In order to obtain the BaP concentration in an easy and timely fashion, a new approach was used to get it indirectly. To investigate the relation between the BaP concentration and total aromatic hydrocarbon concentration, all samples were analyzed by GC/MS. The results showed that the total BaP concentration is significantly correlated with the concentration of particulate-phase aromatic hydrocarbons, and the Pearson correlation coefficient was 0.740. The results of the correlation analysis indicated that the BaP concentration may be predicted based on the aromatic hydrocarbon concentration. A linear regression model was thus established to estimate the BaP concentration. The relationship between the concentrations of BaP and aromatic hydrocarbons was found by curve fitting, and is $0.197\ln(\text{aromatic hydrocarbons}) + 3.405 = \ln(\text{BaP})$. The findings may be applied to a real-time monitoring system for BaP in fugitive emissions.

Keywords: BaP; Aromatic hydrocarbons; Fugitive emissions; Correlation; Coke plant.

INTRODUCTION

An aromatic hydrocarbon is a hydrocarbon with alternating double and single bonds between carbon atoms. It contains monocyclic and polycyclic aromatic hydrocarbon. The toxicity, carcinogenicity and mutagenicity of aromatic hydrocarbon have led to increased concerns about human health. The benzo[a]pyrene (BaP) is the best known for serious carcinogenicity and toxicity (Kirton *et al.*, 1991; Zhu and Matsushita, 1997; Pufulete *et al.*, 2004; Okona-Mensah *et al.*, 2005; Cheng *et al.*, 2010). In the coke industry, coke oven workers are heavily exposed to BaP by inhalation and skin contact. Occupational exposure to BaP has been associated with lung, skin and bladder cancers of humans (Menzie *et al.*, 1992; Boffetta *et al.*, 1997; Aries *et al.*, 2008; Olsson *et al.*, 2010).

Air pollution is a serious problem in Shanxi. The coke industry is considered as a major source of pollution. Statistics suggest that there are about 227 operating coke plants that account for 40 percent of air pollution in the whole province. Coke oven gas emitted during the coke-

making process. Especially, the volatile components of the coal turn into gas, which is hopefully recycled, though some is diffused in the air and called coke oven leakage or fugitive coke oven gas. A great deal of leakage of flue gas is emitted during the process of charging and pushing because of coke exposure when coke oven door is opened, or some misoperation occurs on Larry car. This condition is regarded as fugitive emission or disordered discharge. There are amounts of BaP existing in the workplace of coke oven top. Most researches focused on the USEPA's 16 priority pollutant polycyclic aromatic hydrocarbons (PAHs) which exist in the particulate matters (Cheng *et al.*, 1998; Shen and Liu, 1999; McCready *et al.*, 2000; Qi *et al.*, 2000; Zeng *et al.*, 2002). It is meaningful to investigate gas-phase aromatic hydrocarbons, which may have a negative impact on coke oven workers' health (Bi *et al.*, 2004). So the gas-phase BaP is not ignored in this paper.

The routine methods of determining BaP are as follows: fluorescence spectrophotometry, gas chromatography, high performance liquid chromatography (Huang, 1991; Kershaw, 1996; Yuki *et al.*, 2007; Campo *et al.*, 2009). The analyte was extracted from samples by liquid-liquid extraction, and separated through a chromatographic column. Quantitation was based on peak areas relative to the corresponding internal standards. As routine method is much more time-consuming, complex and expensive, it might not be applied

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broadly, Shanxi attempted to develop a real-time monitoring system for fugitive emission pollution sources which is effective and could be used to monitor the BaP at any time.

The main objective was to find a substitution that has a linear relationship with BaP that can be determined easily. It was considered that the total aromatic hydrocarbons might be easier and faster to be determined. Both the particulate-phase and gas-phase aromatic hydrocarbons were analyzed in this study, in order to investigate the correlation between the BaP concentration and aromatic hydrocarbon concentration. A new approximate calculation formula of BaP concentration might be proposed by using linear regression model. And this result was expected to apply to a real-time monitoring system of pollution sources.

MATERIALS AND METHOD

Sampling

Four coke plants in Shanxi were selected. Samples were obtained from top of coke oven batteries. The sampling point was set between the No. 1 and No. 4 ascension pipe. The samples were collected by an automatic high-volume air sampler for 1.5 h at a flow rate of 180 L/min. A sampling pump was used to draw air through the versatile sampler that would be used to collect both the particulate and gas-phase of aromatic hydrocarbons. A glass fiber filtration (GFF) membrane precedes the front section and a polyurethane foam (PUF) plug followed the back section. The particulate matters were sampled by glass fiber filtration membrane (TFAGF810: 8" × 10" New York, USA) and the gas phase was sampled by polyurethane foam plug (No.TPUF3: 2.5" × 3" New York, USA). The glass fiber filtration membrane has been heated in muffle furnace (XL-1 Hebi, China) at 500°C for 4.5 h and the polyurethane foam has been extracted for 48h at 42°C by using Soxhlet extractor with dichloromethane before sampling. All samples were performed in the fine days in summer during the coke-making process.

Analytical Procedure

The samples were extracted using Soxhlet extraction apparatus with dichloromethane (42°C for 48h) after the addition of surrogate standards (naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, and perylene-d₁₂). The extracts were evaporated in a rotary evaporator then transferred into a fractionation column which was prepared by mixing silica gel and alumina (100–200 mesh Tianjin, China). The fractionation column was eluted with 200 mL of n-hexane. The extracts were subjected to clean-up on silica gel and alumina columns to remove saturated hydrocarbons. After clean-up, the aromatic hydrocarbons fraction was eluted by a further 50 mL dichloromethane at the rate of 2 mL/min. then transferred into weighing bottles. All the weighing bottles were stored in a dry and lucifugal place at 4°C and air-dried till constant weight. The mass of total aromatic hydrocarbons were accurately weighed. The concentrated samples were further concentrated to 1.0 mL using nitrogen evaporator (BF-2000 Beijing, China) and spiked with pyrene-d₁₀ as the internal standard solutions before GC/MS analysis for BaP.

The BaP was analyzed by a gas chromatograph (GC) equipped with a mass selective detector (MSD) (Thermo Fisher, Focus GC/DSQII) and a computer workstation. The capillary chromatographic column (DB-5MS) was 30 m × 0.25 mm i.d. × 0.25 μm film thickness. This GC/MS was operated under the following conditions: injection volume 1 μL, injections were perform in splitless mode at 250°C, ion source temperature at 300°C; oven heating from 50 to 200°C at 15 °C/min, 200 to 310°C at 3 °C/min, and then constant at 310°C for 40min. Quantitation of BaP was performed in the selected ion monitoring (SIM) mode.

Quality Control

Surrogate standards were added to assess possible losses during the analytical procedure. 1 μL of a 4 μg/μL solution of surrogate compounds (naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, chrysene-d₁₂, and perylene-d₁₂) was injected to each sample extraction by using the manual syringe (Hamilton Series 600 Bonaduz, Switzerland). Sample extraction was repeated if surrogate recovery efficiencies were < 75% or > 115%. The total recoveries of aromatic hydrocarbon ranged from 77.2% to 114.2%, averaging 101.7%. Analyses of blank experiments including GFF and PUF revealed no detectable contamination.

Three breakthrough tests were investigated by adding an additional GFF and 1/3 PUF separately during the repeated sampling process. The additional GFF and 1/3 PUF were analyzed individually and compared to examine the aromatic hydrocarbons mass collected. The results showed that no significant aromatic hydrocarbons mass was collected in the additional GFF and PUF.

Duplicate samples were treated as above. The extracts were concentrated to 1 mL and transferred to glass vials with internal standard compounds were added. 1 μL of extracts was analyzed by GC/MS. The residue was transferred into weighing bottles. All the weighing bottles were stored in a dry and lucifugal place at 4°C and then air-dried till constant weight. The residual aromatic hydrocarbons were dissolved after adding dichloromethane into weighing bottles and prepared for GC/MS analysis. 5 species volatile PAHs consisted of fluorene, anthracene, phenanthrene, fluoranthene and pyrene were analyzed individually to determinate the amount of volatile aromatic hydrocarbons loss during the weighing process. It is showed that the loss of volatile PAHs was rather low.

All glassware was cleaned using an ultrasonic cleaner (KQ-100B Kunshan, China) with distilled water and dichloromethane then heated at 500°C for 4.5 h.

Statistical Analysis

The IBM SPSS Statistics Version 19.0 was used as statistical tool: Pearson correlation coefficients were estimated to evaluate the correlation between two variables: BaP and aromatic hydrocarbons levels. In order to find the best-fit model between the aromatic hydrocarbons and the BaP concentrations, linear regression model was used.

In order to evaluate possible differences between each independent sample for each sampling place, student's t-tests were used. Statistical analyses of data from coke oven

gases were performed. In all cases a P-value of < 0.05 was considered significant.

This analysis was based on the assumption that all the samples collected represent a randomly selected and thus representative sample of the population. It is considered that the general correlation existing in samples which collected during an arbitrary time interval in production process is generally true.

RESULTS AND DISCUSSION

BaP Concentrations

The BaP and aromatic hydrocarbons concentrations in the samples are shown in Table 1. Among the four coke plants, the highest average amount of total aromatic hydrocarbons and BaP were observed in the air samples from coke plant D. The lowest concentrate of BaP in airborne particulates was observed in coke plant B. One of the main objectives of this work was to assess the BaP concentrations on the top of coke ovens. Among the samples, the highest average concentration of particulate-phase aromatic hydrocarbon and BaP were observed in Plant D. The highest average concentration of gas-phase aromatic hydrocarbon and BaP were observed in Plant C, and the highest concentration of total BaP and aromatic hydrocarbons were observed in Plant D and Plant C, respectively. Both the two phase aromatic hydrocarbons and BaP concentrations in Plant B were higher than Plant A. The high concentration contaminant might indicate that the coke oven would be sealed bad. It was considered that the concentration of particulate-phase aromatic hydrocarbons tended to be disproportionate to the gas-phase aromatic hydrocarbons concentration.

In fact, there were three samples had exceeded the

maximum accepted level $2.5 \times 10^{-3} \text{ mg/m}^3$ recommended by Ministry of Environmental Protection of the People's Republic of China. The highest concentration of BaP in airborne particulates has been beyond the recommended standard as high as 5.7 times. There are a certain amount of gas-phase aromatic hydrocarbon exist in the fugitive emissions, but it is not limited in the national standard.

Correlation and Linear Regression Analysis

The correlations between two variables were investigated on an individual basis using log-transformed values were showed in Table 2. Through correlation analysis, it was showed that the concentrations of particulate-phase aromatic hydrocarbons were significantly correlated with the particulate-phase BaP ($R = 0.741$, $P = 0.001 < 0.05$). The concentrations of particulate-phase aromatic hydrocarbons were significantly correlated with total BaP ($R = 0.740$, $P = 0.001 < 0.05$). The concentrations of total aromatic hydrocarbons were significantly positively correlated with total BaP which contained both the particle-phase and gas-phase ($R = 0.633$, $P = 0.008 < 0.05$).

The significant correlation between the variables we analyzed came out as we expected. This seems to indicate that the BaP distribution characteristics of aromatic hydrocarbons from each coke plant were the same. The distribution characteristics could be characterized with their physicochemical properties or chemical constitution of coking coal.

Pearson correlation coefficients were greater than 0.6 ($P < 0.05$ in all cases). BaP concentrations were predicted based on aromatic hydrocarbons concentrations by applying linear regression models to the obtained data. The analyses of data were performed after (natural) logarithmic transformation of

Table 1. Aromatic hydrocarbons and benzo[a]pyrene content in the workplace air of coke oven top fugitive emissions.

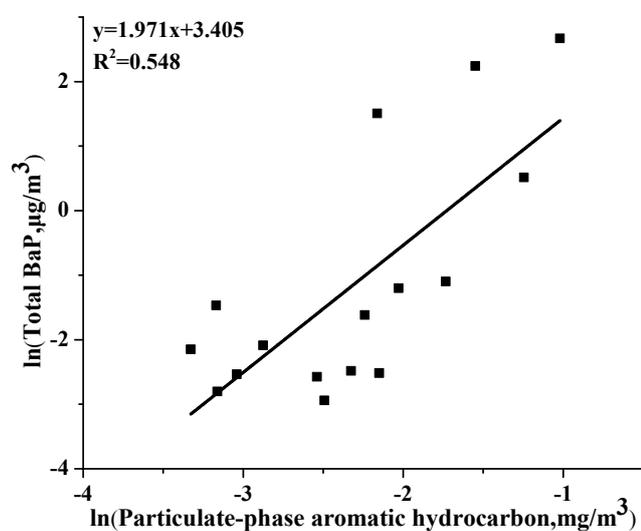
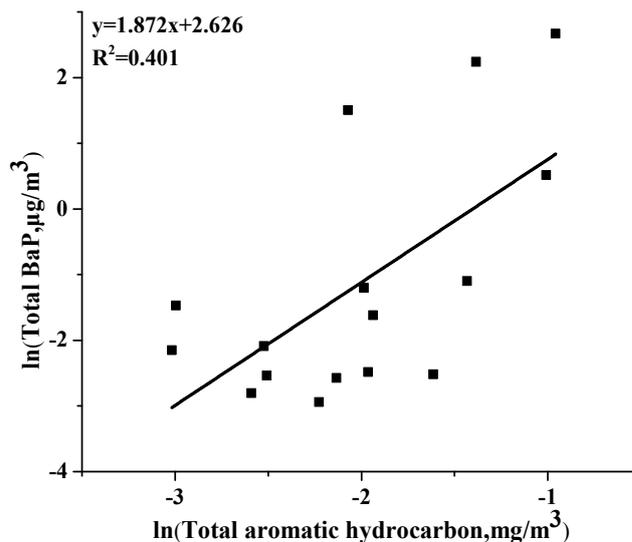
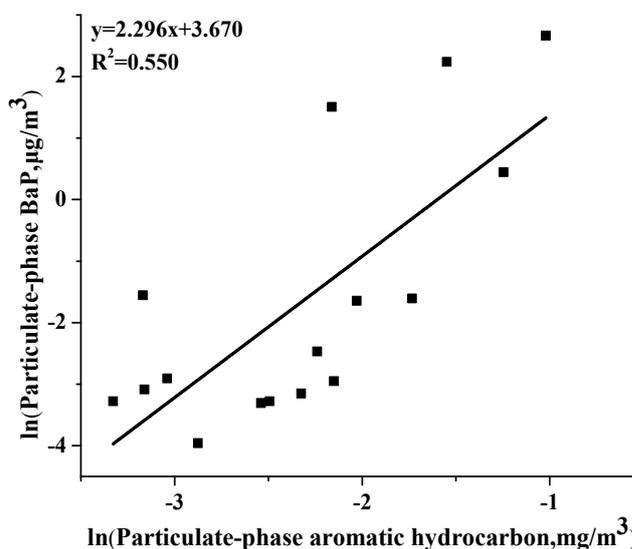
	Particulate phase		Gas phase		Total	
	aromatic hydrocarbon (mg/m^3)	BaP ($\mu\text{g/m}^3$)	aromatic hydrocarbon (mg/m^3)	BaP ($\mu\text{g/m}^3$)	aromatic hydrocarbon (mg/m^3)	BaP ($\mu\text{g/m}^3$)
A-1	0.03587	0.03761	0.01304	0.07913	0.04891	0.11674
A-2	0.04239	0.04565	0.03261	0.01500	0.07500	0.06065
A-3	0.04783	0.05456	0.03370	0.02467	0.08152	0.07923
A-Average	0.04203	0.04594	0.02645	0.03960	0.06847	0.08554
B-4	0.10652	0.08459	0.03760	0.11366	0.14411	0.19825
B-5	0.08271	0.03759	0.02506	0.01529	0.10777	0.05288
B-6	0.09774	0.04273	0.04261	0.04085	0.14035	0.08358
B-7	0.05639	0.01905	0.02381	0.10501	0.08020	0.12406
B-Average	0.08584	0.04599	0.03227	0.06870	0.11811	0.11469
C-8	0.17669	0.20038	0.06203	0.13365	0.23872	0.33402
C-9	0.28759	1.55620	0.07707	0.11466	0.36466	1.67086
C-10	0.13158	0.19267	0.00564	0.10771	0.13722	0.30038
C-11	0.07895	0.03665	0.03947	0.03947	0.11842	0.07612
C-12	0.11654	0.05226	0.08271	0.02857	0.19925	0.08083
C-Average	0.15827	0.40763	0.05338	0.08481	0.21165	0.49244
D-13	0.21256	9.38464	0.03792	0.00268	0.25048	9.38732
D-14	0.04203	0.21140	0.00797	0.01857	0.05000	0.22998
D-15	0.11507	4.50968	0.01101	0.00345	0.12608	4.51313
D-16	0.36074	14.3771	0.02331	0.00507	0.38405	14.3822
D-Average	0.18260	7.12071	0.02005	0.00744	0.20265	7.12816

Table 2. Pearson correlation coefficients between two variables.

	Particulate-phase aromatic hydrocarbons	Gas-phase aromatic hydrocarbons	Total aromatic hydrocarbons
Particulate-phase BaP	R = 0.741 P = 0.001		
Gas-phase BaP		R = 0.150 P = 0.578	
Total BaP	R = 0.740 P = 0.001	R = -0.093 P = 0.732	R = 0.633 P = 0.008

analyte levels to remove heteroscedasticity and satisfy normality assumptions. Simple linear regressions of all subjects gave the following relationships: $0.197\ln(\text{particulate-phase aromatic hydrocarbons}) + 3.405 = \ln(\text{total BaP})$; $1.872\ln(\text{total aromatic hydrocarbons}) + 2.626 = \ln(\text{total BaP})$; $2.296\ln(\text{particulate-phase aromatic hydrocarbons}) + 3.670 = \ln(\text{particulate-phase BaP})$. The linear regression analysis between BaP and other variables were investigated on an individual basis using log-transformed values as illustrated in Figs. 1–3.

The coefficient of determination (R-square) ranges from 0 to 1. An R^2 of 1.0 indicates that the regression line perfectly fits the data. The R^2 ranged around 0.5 in the present linear regression. There are several desirable results but it turned out to be possible to predict the BaP concentration. A multivariate linear regression model is suitable for precisely predicting. In multiple linear regression, there are one or more explanatory variables. It can be thought of an extension of simple linear regression. It is reported that the BaP concentrations in ambient air could be predicted by using Multivariate linear regression models (MLRM). The model was applied to the experimental BaP data based on PM_{10} concentrations. The temperature, the solar radiation and the wind speed were considered in the model (Callén *et al.*, 2010). The temperature of coke oven battery and its surroundings is held in a narrow range. It takes a short time from diffusion of aromatic hydrocarbons leakage to be collected. The

**Fig. 1.** Result of linear regression analysis between the particulate-phase aromatic hydrocarbons and total BaP.**Fig. 2.** Result of linear regression analysis between the total aromatic hydrocarbons and BaP.**Fig. 3.** Result of linear regression analysis between the particulate-phase aromatic hydrocarbons and BaP.

temperature, the solar radiation and other parameters were not considered in this study. Based on multivariate linear regression model, the approximate calculation formula might be revised according to various parameters such as charging mode, the coal blending ratio, coke oven operating

temperature, as well as technological technique and equipment conditions of each plant for the future research.

The concentration of gas-phase aromatic hydrocarbons was insignificantly correlated gas-phase BaP ($R = 0.150$, $P = 0.578 > 0.05$). The total aromatic hydrocarbons concentrations were considered as irrelative to with gas-phase BaP ($R = -0.093$, $P = -0.732 > 0.05$). The significant correlation between gas-phase BaP concentration and the aromatic hydrocarbon concentration did not come out as we had anticipated. Fluorene, phenanthrene and their methylated derivatives, fluoranthene and pyrene were the dominant aromatic hydrocarbon species in the gas phase, while higher ring species PAHs (5 rings and above) dominated in the particulate phase (Bi *et al.*, 2004). This was due to the physicochemical properties of aromatic hydrocarbons. The different conditions of sampling points might lead to the various phase partitions. Samples were obtained from four coke plants in which particulate matters contents, coke oven temperature and atmospheric pressure were not the same. Partial low molecular weight aromatic hydrocarbons, such as naphthalene, acenaphthene or phenanthrene could also adsorb on the particulate matters if the particulate matters concentrations were high at the lower temperature and pressure. The aromatic hydrocarbon is a kind of volatile organic compounds, and some of them might evaporate into the gas phase at a high temperature and pressure. It might give rise to a higher average concentration of particulate-phase aromatic hydrocarbon and BaP in Plant D but a higher average concentration of gas-phase aromatic hydrocarbon and BaP in Plant C. Whereas, BaP and other high molecular weight aromatic hydrocarbons usually mostly attached to the particulate matter. The adsorption or evaporation of the aromatic hydrocarbons might not affect BaP trapping and quantitative analysis of total BaP in this paper

The Aromatic Hydrocarbon Mixture Collection and Determination

The conventional means of particulate-phase aromatic hydrocarbons trapping was time-consuming for real-time monitoring. It is always necessary to choose a proper method to determine aromatic hydrocarbons. Activated carbons or other adsorbent could adsorb aromatic hydrocarbons, which would desorption when the adsorbent was heat up. Li determined nine kinds of aromatic hydrocarbon and halogenated hydrocarbon in ambient air with heat desorption – GC (Li *et al.*, 2004). They tested the effect of absorption and desorption of automatic heat desorption (Tenax TA absorbent). All elements' absorption effect was more than 97%, the average desorption effect was 98.9%. It is a quicker and reliable method than any before it.

Thomas researched fluorescence spectra of aromatic hydrocarbon molecules in the gas phase for comparison with Blue Luminescence emission (Thomas and Witt, 2006). It was demonstrated that aromatic hydrocarbons could be responsible for the Blue Luminescence. The BL was more likely due to a mixture of small neutral monomer aromatic hydrocarbons but not a single aromatic hydrocarbon. Their luminescence was easier to detect because of their higher

fluorescence efficiencies. It seems that the fluorescence spectra apply to determination of mixture of aromatic hydrocarbons. An effective method based on fluorescence spectrophotometry might be developed to estimate the BaP concentrations fast and easily.

CONCLUSIONS

The concentrations of aromatic hydrocarbons and BaP in the fugitive emissions at workplace of coke oven top had been studied. A higher BaP concentration was founded in the fugitive emissions. Several samples had exceeded the maximum accepted level. It was necessary to monitoring the BaP concentration in real time.

The present results showed that the particulate-phase aromatic hydrocarbons concentrations were significantly positively correlated to total BaP concentrations. A statistical model based on linear regression was studied to predict concentrations of BaP in the fugitive emissions. This proposed model had got good robustness and predictability.

Moreover, particulate-phase or total aromatic hydrocarbons may act as an indicator compound of BaP in fugitive emissions. The approach to determine BaP concentration indirectly which is based on the aromatic hydrocarbons might take a very short time and be inexpensive. It might be applied to a real-time monitoring system of BaP from the fugitive emissions in the future.

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