Printed in the Republic of Korea ANALYTICAL SCIENCE
& TECHNOLOGY Vol. 31 No. 1, 14-22, 2018

https://doi.org/10.5806/AST.2018.31.1.14

Mainstream smoke level of harmful substances in korean domestic cigarette brands

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(Received December 4, 2017; Revised January 30, 2018; Accepted January 31, 2018)

Abstract After signing the WHO FCTC in 2003, South Korea ratified the FCTC in 2005. This study was conducted to provide data on toxic constituents that can be used as useful information for the level of exposure to Korean smokers. Emissions data from five brands of cigarettes were tested under the ISO and "Canadian Intense (HCI)" smoking regimes, respectively. We conducted an analysis of 25 compounds containing nicotine, tar, carbonyls, phenolics, volatile organic compounds (VOCs), and semi-VOC cigarette smoke. Tar and nicotine showed levels of 4.3 to 5.8 mg/cig and 0.4 to 0.5 mg/cig, respectively, which are within the range of tolerance presented in ISO 8243. In the case of carbonyls, formaldehyde was detected within a range of 8.2 to 14.3 μ g/cig, and acetaldehyde was present within a range of 224.7 to 327.2 μ g/cig under the ISO smoke regime. Crotonaldehyde was not detected under the ISO regime, and all of the carbonyls showed values 2.3 to 4.5 times higher under the ISO regime and 117.5 to 184.7 μ g/cig under the ISO regime and 221.0 to 377.0 under the HCI regime. To summarize, most of the constituents showed a tendency to be detected at levels 2 to 4 times higher under the HCI regime than under the ISO regime. Above all, these results represent the first analysis in Korea from an independent institute of tobacco companies under accreditation of ISO 17025.

Key words: tobacco, hazard constituents, constituents analysis, FCTC

1. Introduction

The World Health Organization's Framework Convention on Tobacco Control (FCTC) is the world's first international public health treaty. The provision needs to be specified now since the FCTC has come into force. The issue of how to test and regulate conventional cigarettes represents a critical challenge for tobacco control.¹ Article 9 of the WHO FCTC deals with the test and regulation of tobacco product contents and emissions, while article 10 deals with disclosure of information about them.

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To date, the primary means of testing cigarette toxicity have been to smoke the cigarettes according to a standard puffing regime and to measure the chemical emissions in the mainstream smoke.¹ A recent study examined the extent to which nicotine emissions from the ISO regime and the Health Canada intense (HCI) regime were associated with nicotine uptake among a sample of Canadian smokers. A commonly used mainstream smoking protocol is the ISO protocol.² The HCI smoking machine protocol was developed to characterize more intense smoking behavior by increasing puff volume, shortening inter-puff interval (IPI), and blocking ventilation holes compared to the ISO protocol.³

Carbonyls, Phenolic, and some volatile organic compounds are potent carcinogens in laboratory animals. The accumulated scientific evidence eventually led to the classification of these compounds as human carcinogens by the International Agency for Research on Cancer (IARC).⁴ Tobacco smoke is an exceedingly complex matrix consisting thousands of compounds. So far, more than 4,000 chemicals have been identified in tobacco and more than 50 of the chemicals are known carcinogens.⁵

In this study, we characterize mainstream smoke of 5 domestic cigarette brand varieties smoked by the ISO and HCI protocols on smoking machines by analytical methods capable of measuring 25 substances (of which 13 are listed by the FDA as HPHCs and Hoffmann lists). This study is the first meaningful data in terms of 25 toxic constituents in mainstream smoke in Korean domestic tobacco products. Five brand varieties are selected considering the five topselling products in 2013. While sampling was usually done on a regional basis in other researches, this study can be representative of data because sampling was done on a nationwide basis. By including products that encompass the majority of the cigarette market, these data will provide useful information when evaluating the level of exposure to Korean smokers. Moreover, this study assessed the level of tobacco emissions in Korean domestic products by comparing it with the results of other countries' analysis.

2. Experimental

2.1. Cigarette sampling

Five commercial cigarette products were collected according to ISO 8243:2013 from Korean retails all over the country.⁶ The cigarette samples showed tar values on the package which were product A at 5.0 mg/cig, product B, D at 6.0 mg/cig, product C at 5.5 mg/cig, product E at 4.5 mg/cig under ISO smoking regime. In order to represent the domestic market, we selected 5 products which have a large sale and similar tar content to each other. While product A, B, C, and D were regular cigarettes, product E was a slim cigarette. A research cigarette, 3R4F (University of Kentucky, Lexington, NY) was included to compare the level of toxicants based on A. Eldridge *et al.*, 2015's research data.⁷

2.2. Cigarette preparation

Cigarettes were conditioned in an environmental chamber held at (22 ± 2) °C and (60 ± 3) % humidity for at least 48 hr prior to analysis using method ISO 3402:1999.⁸ Cigarettes smoked according to ISO 3308:2000² and HCI protocols using the equipment. Cigarettes were smoked under International Organization for Standardization (ISO; 60s interval, 35 ml puff volume, and 2s puff duration) and Health Canada Intense (30s interval, 55 ml puff volume, 2s

Table 1. Parameters of ISO and HCI regimes

Smoking regimen	Puff volume (ml)	Puff frequency	Ventilation holes		
ISO 3308:2000, Routine analytical cigarette-smoking machine — Definitions and standard conditions	35	Once every 60 s	No modifications		
HCI Same as ISO 3308:2000 but modified as indicated.	55	Once every 30 s	All ventilation holes must be blocked with Mylar adhesive tape.		

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puff duration, and blocked filter ventilation holes) conditions (*Table* 1).⁹ All cigarette samples were smoked using a 20-port (rotary and linear, depending on constituent being examined). Method accuracy was determined by analysis of reference cigarettes 1R5F and CM7 and the results were compared with CORESTA collaborative study.¹⁰

2.3. Analysis procedure

2.3.1. Tar, nicotine, and water

Tar, nicotine and water were determined according to the relevant ISO standards (ISO Standard 3308: 2012; ISO Standard 4387:2000; ISO Standard 8454: 2007; ISO Standard 10315:2013; ISO Standard 10362-1:1999).^{2,11-13} Nicotine and water were determined from isopropanol extracts of total particulate matter (TPM) retained by the smoke collection pad using gas chromatography (GC). Tar was calculated from the weight of TPM less the weights of water and nicotine.

2.3.2. Hydrogen cyanide

In the case of hydrogen cyanide, Smoke was collected on CFPs in combination with either one or two impingers, derivatised with 0.1N NaOH (sodium hydroxide) in preparation for continuous flow analysis according to Health Canada official method T-107, 1999.¹⁶

2.3.3. Selected carbonyls

Selected carbonyls (Methyl ethyl ketone, Acetaldehyde, Acetone, Acrolein, Butyraldehyde, Crotonaldehyde, Formaldehyde, Propionaldehyde) were determined according to Health Canada official method T-104, 1999.¹⁴ Carbonyls in mainstream smoke were trapped by two impingers with DNPH solution and the impinger solution was analyzed by high-performance liquid chromatography-UV/VIS detector (HPLC-DAD)

2.3.4. Phenolic compounds

Phenolic compounds (phenol, 3 cresol isomers, hydroquinone, catechol, and resorcinol) were obtained to consistent results by liquid chromatographyfluorescence detector (LC-FLD) after extracting with 1 % acetic acid in water collected "tar" on a Cambridge filter pad (CFP). The compounds were determined according to Health Canada official method T-114.¹⁵

2.3.5. Volatile organic compounds

Volatile organic compounds (VOCs, 1,3-Butadiene, Isoprene, Acrylonitrile, Benzene, Toluene, Chloride) in mainstream smoke were collected using two impinger solution at -70 °C with methanol and analyzed by GC-MS. The VOCs were determined according to Health Canada official method T-116, 1999.¹⁷

2.3.6. Semi volatile organic compounds

The studied analytes of selected semi-volatile organic compounds were pyridine; quinoline and styrene. The compounds were trapped by two impinger solution at -70 °C with methanol and CFP. The CFP were extracted by impinger solution and then analyzed by GC-MS according to Health Canada official method T-112, 1999.¹⁸

3. Results

3.1. Emission levels of nicotine, water and tar in mainstream smoke

Levels of nicotine, water and tar in mainstream smoke are presented in *Table* 2. The amount of nicotine in all brands averaged 0.45 (\pm 0.09) mg/cig for ISO regime and 1.27 (\pm 0.28) mg/cig for HCI regime. The results of water content in mainstream smoke of cigarette brands were 1.27 (\pm 0.99) mg/cig for ISO regime and 2.49 (\pm 1.61) mg/cig for HCI regime. As for tar contents, the results were determined to 5.05 (\pm 0.75) mg/cig for ISO regime. Relative nicotine, water and tar levels generated from the different cigarette brand between the two smoking regime are compared in *Table* 2. The ratio of HCI regime to ISO regime showed 2.5 ~ 3.0.

3.2. Emission levels of hydrogen cyanide in mainstream smoke

In case of hydrogen cyanide (HCN), there were two kinds of trapping system. One was to use pad and the other was to use impinger. The total results were determined by summation of two trappings. *Table* 2 presents the results of HCN in mainstream smoke of five commercial cigarette brands. Overall, the pad trapping system collected more amount of HCN compared to impinge system except for product B. In particular, product B showed the highest amount of HCN among five cigarette brands and HCN amounts by the pad and impinger trapping were similar as 10.8 µg/cig and 13.0 µg/cig for ISO regime and 40.2 µg/cig and 45.1 µg/cig for HCI regime, representatively. Total HCN showed 19.9 (±4.0) µg/cig for ISO regime and 66.2 (±19.2) µg/cig for HCI regime. The ratio of HCI regime to ISO regime ranged from 2.3 to 3.6.

3.3. Emission levels of selected carbonyls in mainstream smoke

The results of the selected carbonyls in cigarette smoke are summarized in Table 2. The ranges of formaldehyde were 11.3 (±3.1) µg/cig for ISO regime and 39.9 (±14.4) µg/cig for HCI regime. The product B had relatively higher levels of formaldehyde (14.3 μ g/cig for ISO regime and 54.2 μ g/cig for HCI regime) compared to other products. The ratio of ISO regime to HCI regime for formaldehyde ranged from 3.1 to 4.2. Acetaldehyde, the compound present in the highest amount among carbonyls, was with a range of 276.0 (±51.3) µg/cig for ISO regime and 729.8 (±134.9) µg/cig for HCI regime. The ratio of ISO regime to HCI regime of Acetaldehyde showed from 2.5 to 3.0. The product B contained the lowest level of acetaldehyde in comparison with other products under both smoke regimes. In case of acetone, the amount of the analyte was determined about 116.0 $(\pm 11.5) \mu g/cig$ for ISO regime and 280.9 $(\pm 39.3) \mu g/cig$ cig for HCI regime. Similar to the acetaldehyde, The ratio of HCI regime to ISO regime was in a range from 2.5 to 3.0. The range of acrolein was $10.1 (\pm 1.3)$ µg/cig for ISO regime and 36.7 (±3.6) µg/cig for HCI regime. The results of ratio of HCI regime to ISO regime showed from 2.9 to 3.8.

As for propionaldehyde, the amount of analyte showed 21.6 (\pm 4.2) µg/cig under ISO regime and 60.8 (\pm 11.7) µg/cig under HCI regime. Unlike other

carbonyls, crotonaldehyde were not detected except for product E under ISO regime. Moreover, its amounts of crotonaldehyde under HCI regime were lower than other products. Overall, The ratio of HCI regime to ISO regine for carbonyl contents was presented in a range from 2.5 to 4.5. Selected carbonyls in mainstream smoke of five cigarette brands showed that there was no apparent trend according to the products.

3.4. Emission levels of phenols in mainstream smoke

The results in *Table* 2 include phenolic compounds in mainstream smoke of five commercial cigarette brands. Hydroquinone was determined in a range of 19.5 (±4.0) µg/cig for ISO regime and 45.3 (±9.0) µg/cig for HCI regime. The highest amount was that of product C and the lowest amount was that of product E under both of regimes. Among the phenolic compounds, catechol showed the highest amount with a range of 63.8 (±16.8) µg/cig for ISO regime and 151.1 (±33.6) µg/cig for HCI regime. O-cresol showed the lowest level among phenolic compounds with value of 1.6 (±0.7) µg/cig for ISO regime and 5.3 (±1.7) µg/cig for HCI regime. Overall, The ratio of HCI regime to ISO regime ranged with 2.0 ~ 4.5.

3.5. Emission levels of volatile organic compounds (VOCs) in mainstream smoke

Table 2 presents volatile organic compounds (VOCs) resulted from five cigarette brands in mainstream smoke. The result of 1, 3-butadiene in all brands showed in range of 20.57 (\pm 5.6) µg/cig for ISO regime and 58.4 (±13.7) for HCI regime. In terms of ISO regime, 1, 3-butadiene in product D showed the highest amount as 26.1 µg/cig but as for HCI regime, the product C included the highest its amount as 72.1 μ g/cig. The amounts of Isoprene were 125.0 (±33.3) µg/cig for ISO regime and 299.0 (±78.0) µg/cig for HCI regime. Under both regimes, the product B showed the lowest level of Isoprene but the product D indicated the highest content. Acrylonitrile was detected from N.D (not detection) to 2.4 µg/cig under the ISO regime. Under the HCI regime, acrylonitrile was detected the 9.55 (± 2.75) µg/cig. In addition, the ratio of contained acrylonitrile between two smoking regimes was lower than other VOCs compounds overall products as $4.5 \sim 10.9$. The amount of benzene averaged $18.4 (\pm 5.4) \mu g/cig$ in terms of ISO regime and $18.7 (\pm 4.35) \mu g/cig$ in case of HCI regime, therefore, the ratio between two regimes was almost 1. The result of toluene was $29.2 (\pm 6.75) \mu g/cig$ under ISO regime and $99.6 (\pm 13.4) \mu g/cig$ under HCI regime. The amounts of toluene in HCI regime were $3.0 \sim 4.4$ times more than those in ISO regime.

3.6. Emission levels of semi volatile organic compounds (pyridine, styrene, quinoline) in mainstream smoke

Table 2 indicated the three compounds in mainstream

smoke of 5 commercial products. Detected Pyridine averaged 1.6 (±0.6) µg/cig under the ISO regime and 8.5 (±2.4) µg/cig under the HCI regime. Ratio between two regimes ranged 3.3 in product B and 6.2 in product E in terms of pyridine. The results of styrene were determined as 1.3 (± 0.5) µg/cig for ISO regime and and 6.6 (±1.3) µg/cig for HCI regime in overall products. Range of ratio for styrene was yielded from 3.7 to 6.6. In case of quinoline, the lowest level among the semi VOCs compounds showed in the results. Detected amounts of quionline were 0.2~0.3 µg/cig under ISO regime and 0.6~0.8 µg/cig under HCI regime, respectively. The ratio between two regimes showed from 2.3 to 3.0. Overall, the product B indicated the lowest level of ratio between two regimes while the product E was the highest level of that.

Table 2. Analyzed data of Korean domestic brands A-E in mainstream smoke

		Unit	ISO regime				HCI regime					
		Unit	А	В	С	D	Е	А	В	С	D	Е
Nicotine		mg/cig	0.5	0.5	0.5	0.5	0.4	1.5	1.0	1.3	1.4	1.0
Tar(NFDFM)		mg/cig	5.7	4.3	5.8	5.1	4.6	18.1	11.1	15.9	16.9	15.6
Water		mg/cig	0.4	2.3	0.3	0.8	0.5	0.9	4.1	1.1	1.3	0.9
HCN		µg/cig	15.9	23.8	21.1	22.2	18.3	47	85.3	51	52.1	50.5
Carbonyls	Acrolein	µg/cig	8.8	10.8	9.1	11.3	11.4	33.1	40.2	33.2	33.2	33.7
	Acetone	µg/cig	106.7	105.5	104.5	123.3	127.4	301.6	241.6	316.1	320.2	319
	Acetaldehyde	µg/cig	257.1	224.7	291.6	324.4	327.2	759.5	594.9	855.6	864.7	832.2
	Formaldehyde	µg/cig	8.4	14.3	10.8	8.2	8.4	35.6	54.2	37.6	25.5	35.2
	Butyraldehyde	µg/cig	14.8	13.8	14.2	19.2	19.5	53.5	43.9	55.7	60.2	53.2
	MEK	µg/cig	22.3	19.6	19.8	24.1	22.4	86.5	62	90	93.2	94.6
	Crotoaldehyde	µg/cig	0	0	0	0	0.9	26.8	26.5	27.4	29	29
	Propionaldehyde	µg/cig	19.8	17.4	19	25.4	25.7	64.8	49.1	66.3	72.4	70.4
Phenols	o-cresol	µg/cig	2	2.3	2.2	1.7	0.9	6.9	5.4	4.7	4.8	3.6
	m+Pcresol	µg/cig	2.5	2.7	2.7	2.1	1	8.8	6.9	6.4	6.3	4.5
	Phenol	µg/cig	8.3	9.7	9	6.5	3.1	28.8	23.4	19.1	18.8	12.6
	Catechol	µg/cig	68.9	70.8	80.5	61.7	47	184.7	139.1	180.9	147.1	117.5
	Resorcinol	µg/cig	2.2	2.4	3.1	2.1	1.3	6.7	5.6	9.4	6.1	4.4
	hydroquinone	µg/cig	21.1	19.8	23.5	21.3	15.5	53.9	36.8	54.2	51.9	36.3
VOCs	Toluene	µg/cig	31.6	28.7	22.4	35.9	28.4	113	86.2	98.6	107	112
	Benzene	µg/cig	21.6	13	16	23.8	19.1	21.1	14.3	20.1	21.4	23
	Acrylonitrile	µg/cig	1.1	0	0.8	2.4	1.2	8.4	6.8	8.2	10.7	12.3
	Isoprene	µg/cig	138.7	91.7	112	158.3	112.3	332	221	369	377	339
	1,3-Butadiene	µg/cig	15	19.2	23.8	26.1	16.4	59.4	44.7	72.1	59.2	59.1
Semi-VOCs	Styrene	µg/cig	1.4	1.8	1.6	1.1	0.8	7.5	6.7	7.8	7.2	5.3
	Quinoline	µg/cig	0.3	0.3	0.3	0.2	0.2	0.8	0.7	0.7	0.6	0.6
	Pyridine	µg/cig	1.8	2.2	2.1	1.7	1	10.7	7.3	10.9	10.3	6.2

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4. Discussion

4.1. ISO and HCI smoking condition

According to A. Eldrige *et al.* (2015)⁷, current and proposed regulations for measurement and reporting or mandated lowering of smoke toxicant levels require data generated at various regimes. In Brazil, ANVISA requires measurement and reporting under the ISO smoking regime. In the USA, the FDA requires toxicant levels under both regimes which are ISO and HCI regimes. With regard to Canadian regulation, every tobacco manufacturer needs to submit data on 41 mainstream smoke emissions for cigarettes sold in the Canadian market to the Minister of Health. The emission data must be produced under two difference regimes, ISO and HCI regimes. Even though the regulation and law in terms of tobacco toxicants have been increased over the world, so far there is only regulation that regulates manufacturers to declare tar and nicotine contents in cigarette under the ISO regime in Korea. Moreover, in Korea

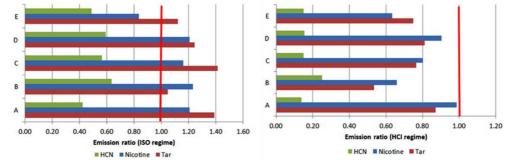


Fig. 1. Emission ratio of HCN, Nicotine, and Tar in mainstream smoke from Korean domestic brands A-E under the ISO and HCI regimes.

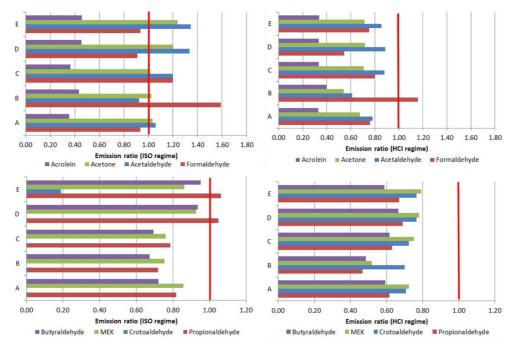


Fig. 2. Emission ratio of selected carbonyls in mainstream smoke from Korean domestic brands A-E under the ISO and HCI regimes.

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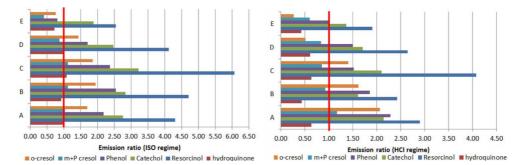


Fig. 3. Emission ratio of Phenols in mainstream smoke from Korean domestic brands A-E under the ISO and HCI regimes.

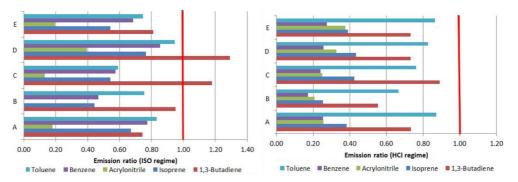


Fig. 4. Emission ratio of VOCs in mainstream smoke from Korean domestic brands A-E under the ISO and HCI regimes.

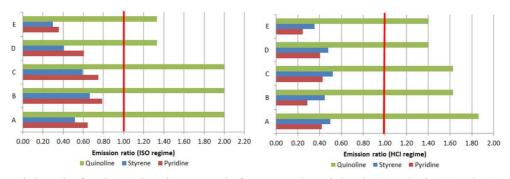


Fig. 5. Emission ratio of semi VOCs in mainstream smoke from Korean domestic brands A-E under the ISO and HCI regimes.

there have not been any official data of toxicants in commercial cigarettes especially under the two smoking regimes.

In this study, HCI smoking regime generally produced more smoke constituent yields than ISO smoking regime. The ratio of two regimes was indicated from 1.0 to 10 and this result was similar to the results of A. Eldrige *et al.* (2015) D.Y. Pazo, F. Moliere, M.M. Sampson *et al.* (2016),^{3,7} which were data of toxicants in the USA, Germany, and the UK, however, there was variation according to the products and substances.

According to the overall results, the results of HCI regimes emitted high amounts of toxicants compared to those of ISO regimes. Mainstream smoke yields as measured by smoking machines increase as larger puff volumes, more frequent puffs are taken, or filter vent-blocking is applied to highly ventilated cigarettes. This is largely the result of more tobacco being consumed during a puff to form mainstream smoke. Many possible changes may occur within the burning cigarette as the cigarettes are smoked with different smoking regimes.²⁰

4.2. Comparison with reference cigarette

As the following figures, each toxicant was compared to data of a product which contained similar tar level (4.4 mg/cig) of the UK commercial cigarette brand based on Eldridge, A., et al., 2015's data. When the data was the reference, the ratios of nicotine and tar were higher than 1 except for nicotine of product E and HCN was less than or around $0.4 \sim 0.6$ under the both of regimes in mainstream smoke of 5 products. In the case of carbonyls, the ratio of acrolein to reference of all products indicated less than 0.5 and crotonaldehyde showed the lowest ratio than other carbonyls which presented around or over 1 ratio in both of regimes. Product B indicated that formaldehyde was the highest ratio in both of regimes to comparison with other products.

Phenols showed distinctive results with other toxicants because other toxicants showed the ratio less or similar with 1 while phenols indicated special higher ratio than reference data. In ISO regime, all products indicated higher ratio than 1.5 with respect to catechol and resorcinol. And product A, B and C showed the ratio of phenol over than 1. The results in HCI regime were similar to those in ISO regime. In addition, product A, B, and C showed the ratio of o-cresol over than 1. In both regimes, resorcinol of product C was the highest ratio as over than 4.0.In terms of quinoline, the result also showed similar to that of phenol compounds. All products indicated over 1 ratio compared to reference cigarette in both of regimes.

According the results of M. Siu, N. Mladjenovic, and E. Soo,¹⁹ the increase of phenols was impacted by ventilation which was believed to lower the smoke temperature at filter interface in the vented filter and shifted the partition of semi-VOCs like phenols towards the particulate phase; however, only ventilation was not determined with respect to increasing of phenols. Moreover, the cigarette samples could not be homogeneous materials in spite of sampling method, ISO 8243:2013.⁶ Therefore, various factors must be considered to reduce the toxicants in mainstream smoke.

5. Conclusion

Since South Korea ratified the FCTC in 2005, to implement Article 9, the testing and measuring of tobacco product contents and emissions, and Article 10, the disclosure of information about tobacco product contents and emissions, it has been necessary to list up data of toxicants. So far only tobacco companies have produced lists with respect to toxicants analyzed by tobacco components in Korea; therefore, it has been difficult to disclose those.

In this study, we analyzed constituents in mainstream smoke in 5 domestic cigarette brand varieties smoked with the ISO and HCI protocols on smoking machines by analytical method capable of measuring 25 substances which are listed by the FDA as HPHCs and Hoffmann lists.

The amount of nicotine in all brands averaged 0.45 (± 0.09) mg/cig for ISO regime and 1.27 (± 0.28) mg/cig for HCI regime. Tar contents were determined to 5.05 (± 0.75) mg/cig for ISO regime and 14.6 (± 3.50) mg/cig for HCI regime. Acetaldehyde was presented as the highest amount among carbonyls and was with a range of 276.0 (± 51.3) µg/cig for ISO regime and 729.8 (± 134.9) µg/cig for HCI regime. Among the phenolic compounds, catechol was the highest amount with a range of 63.8 (± 16.8) µg/cig for ISO regime and 151.1 (± 33.6) µg/cig for HC regime.

Total HCN showed 19.9 (±4.0) µg/cig for ISO regime and 66.2 (±19.2) µg/cig for HCI regime. The amounts of Isoprene were 125.0 (±33.3) µg/cig for ISO regime and 299.0 (±78.0) µg/cig for HCI regime. Pyridine averaged 1.6 (±0.6) µg/cig under the ISO regime and 8.5 (±2.4) µg/cig under the HCI regime. In comparison with reference data in an UK commercial cigarette, the ratio of catechol, resorcinol, phenol, and quinoline were indicated around 2.0 and there was variation according to the products.

Above all, the results were the first toxicants analysis of commercial cigarette in Korea independent of tobacco companies under ISO 17025 accreditation. By including products that encompass the majority of the cigarette market, these data will provide useful information when evaluating the level of exposure to Korean smokers.

Acknowledgements

This research was supported by the Ministry of Food and Drug Safety grant number (14182MFDS977) in 2015.

Competing interests: None to declare.

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