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基于车载测试的重型柴油车尾气典型烷烃排放特征

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摘要: 本文采用车载排放测试系统对 11 辆国 I ~ 国 IV 标准重型柴油车进行实际道路测试, 利用 GC-MS 对样品中典型烷烃进行定量分析, 解析重型柴油车尾气典型烷烃排放特征及规律. 结果表明, 排放标准对重型柴油车尾气中正构烷烃、藿烷类有机物排放有显著影响, 总体呈现随排放标准的加严而降低的趋势, 相比于国 I 测试车辆, 国 IV 测试车辆正构烷烃、 $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C30 藿烷 (C30-藿烷)、22S-和 22R- $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C31 升藿烷 (22S-C31 升藿烷; 22R-C31 升藿烷) 总排放因子分别降低了 72.23%、64.95%、70.78% 和 74.68%. 气相正构烷烃呈双峰前锋型, 以 C17 ~ C18 为主峰碳, 固相呈单峰前锋型, 以 C18 ~ C21 为主峰碳. 藿烷类有机物其 22S-C31 升藿烷 / (22S-C31 升藿烷 + 22R-C31 升藿烷) 的比值在 0.46 ~ 0.56 之间, 平均值为 0.50, 符合石油中藿烷的分布特征. 正构烷烃总排放因子与 $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C30 藿烷总排放因子呈现出一定的线性关系, 其 R^2 为 0.9268. 此外, 行驶工况对测试车辆正构烷烃及藿烷类有机物排放有较大影响, 非高速工况下排放因子是高速工况的 1.69 ~ 2.42 倍.

关键词: 重型柴油车; 车载排放测试系统 (PEMS); 正构烷烃; $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C30 藿烷; 22R- $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C31 升藿烷; 22S- $17\alpha(\text{H})$ 、 $21\beta(\text{H})$ -C31 升藿烷

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Exhaust Emission Characteristics of Typical Alkanes from Heavy-Duty Diesel Vehicles Based on a Portable Emission Measurement System

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Abstract: The on-road emissions of typical alkanes from 11 heavy-duty diesel vehicles with different emission standards (from China I to China IV) were tested using a portable emission measurement system (PEMS) and quantified by gas chromatography - mass spectrometry (GC-MS). Our aim was to analyze the emission characteristics of typical alkanes in heavy-duty diesel vehicle exhaust. The results show that the emission standard significantly affected the emission factors (EFs) of *n*-alkanes and hopanes. Vehicles with higher emission standards had lower EFs. Compared with China I vehicles, the total EFs of *n*-alkanes, $17\alpha(\text{H})$, $21\beta(\text{H})$ -C30 hopane (C30-hopane), and 22S- and 22R- $17\alpha(\text{H})$, $21\beta(\text{H})$ -homohopane (22S-C31 and 22R-C31 homohopane) from China IV vehicles were significantly reduced by 72.23%, 64.95%, 70.78%, and 74.68%, respectively. The peak carbon numbers of gaseous *n*-alkanes were 17 to 18, while they were 18 to 21 in particulate *n*-alkanes. The 22S-C31 homohopane / (22S-C31 homohopane + 22R-C31 homohopane) ratios ranged from 0.46 to 0.56, with an average of 0.50, which conform to the characteristics of hopanes in petroleum. The total EFs of *n*-alkanes had a good linear relationship with the total EFs of C30-hopane, and the R^2 was 0.9268. Furthermore, the driving conditions had a great influence on the emissions of *n*-alkanes and hopanes. Specifically, the EFs of *n*-alkanes and hopanes on non-highway roads were 1.69 to 2.42 times greater than those on highways.

Key words: heavy-duty diesel vehicles; portable emission measurement system (PEMS); *n*-alkanes; $17\alpha(\text{H})$, $21\beta(\text{H})$ -C30 hopane; 22R- $17\alpha(\text{H})$, $21\beta(\text{H})$ -homohopane; 22S- $17\alpha(\text{H})$, $21\beta(\text{H})$ -homohopane

随着机动车保有量的持续增长, 机动车尾气排放已成为我国城市地区大气污染的主要来源^[1,2]. 机动车尾气中包括很多对人体和大气环境有毒有害的物质, 如多环芳烃、烷烃以及许多其他有机物^[3,4], 其中以烷烃类物质为主. 研究表明正构烷烃是机动车尾气烷烃类物质中可定量的优势组分^[5], 其对人体的毒性表现为窒息作用、对中枢神经系统的麻醉作用和对皮肤黏膜、呼吸道的刺激作用等^[6]. 正构烷烃对人体的危害程度会随碳数的增加

而增大, 当碳数大于 16 时, 不仅能损伤皮肤, 甚至产生皮肤癌^[7]. 重型柴油车虽然在燃油车总保有量中比例较小, 但其污染物排放量却远远高于其他轻型车. Grosjean 等^[8]研究表明, 重型车各种污染物排放因子均是轻型车几倍至几十倍以上. 研究重型柴

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油车尾气烷烃类物质排放特征对大气污染的源解析以及降低对人体健康危害有着重要意义。

目前国内外研究机动车尾气烷烃类物质多采用台架测试和隧道测试^[9,10]。但台架测试不能反映机动车实际道路行驶中真实排放情况,可能会严重低估机动车的排放^[11]。而隧道测试只能得到单一工况下测试车辆综合排放因子,并且测试过程中会受到环境背景值以及气候条件的影响^[12,13]。二者均不能很好地反映实际道路机动车排放的真实水平。随着测试技术的不断发展,车载排放测试逐渐成为机动车排放测试的研究热点^[14~16],该方法可以获得机动车实际道路行驶过程中车辆各项行驶状态参数、污染物浓度以及外界环境参数,能够很好地反映单车的工况对瞬时污染物排放的影响^[17,18]。目前采用车载测试研究重型柴油车尾气烷烃类有机物的研究相对较少,因此本研究基于车载测试系统研究重型柴油车尾气典型烷烃排放特征及关键因素的影响,以期为我国重型柴油车污染控制提供数据支撑。

1 材料与方法

1.1 测试系统

本研究车载排放测试系统主要包括三大部分: 气态污染物测试系统(SEMTECH-DS)、等比例稀释

系统(SEMTECH-MPS)和烷烃采样系统。分别利用石英膜和聚氨酯泡沫(PUF)对颗粒态和气态烷烃进行采样,PUF采用多级串联形式吸附。采样流量控制在 $5\text{ L}\cdot\text{min}^{-1}$,每次采样前后对采样系统进行气体流量校正。

1.2 测试路线

本研究测试路线涵盖非高速路和高速路,分别代表非高速工况和高速工况。非高速测试路段以苏州浒墅关装卸运输公司为起点,东桥收费站为终点,全长 14.6 km ;高速路段以东桥收费站为起点,以东山收费站为终点,全长 32.4 km 。测试期间所有测试车辆为空载,测试均处于热启动状态,为确保测试采集样品在离线分析时不低于仪器的检测下限,测试采样时间在 $30\sim 40\text{ min}$ 。

1.3 测试车辆

本研究选取11辆在用重型柴油车(总质量大于 $12\,000\text{ kg}$),包括国I~国IV这4种排放标准,每种标准测试车辆2~3辆。其中国I至国III标准重型柴油车未安装任何尾气处理装置,而国IV标准重型柴油车全部安装了选择性催化还原尾气处理装置(SCR)。所有测试车辆均由运输公司租赁,发动机在生产时都符合相关标准且运行良好,尿素是SCR正常运转工作的必要条件,测试车辆SCR装置均正常加入尿素。车辆详细信息如表1所示。

表1 测试车辆详细信息

Table 1 Detailed information concerning the test vehicles

编号	技术	生产年份	车辆型号	发动机号	总重/kg	额定功率/kW	发动机排气量/L	累计行驶里程/km
1	国 I	2003	解放 CA1113P1K2L8	50374921	15 880	165	4.75	318 041
2		2001	永旋 HYG9160	00430005	36 305	193	9.73	245 683
3		2007	东风 EQ4181W	69143470	39 500	191	8.8	177 234
4	国 II	2007	东风 EQ1143ZE	E0246700165	14 000	100	4.26	292 109
5		2007	解放 CA1128PK2L2A80	50757114	12 175	100	4.75	161 909
6		2010	解放 CA4206P1K2T3EA80	51681139	34 015	203	7.70	88 000
7	国 III	2009	解放 CA1133P9K2L4E	51406342	13 470	101	4.75	220 813
8		2009	解放 CA1123P9K2L4E	52385597	12 015	106	4.75	213 518
9		2015	解放 CA1160P62K1L3E4	60322838	15 585	118	4.04	3 582
10	国 IV	2015	解放 CA1310P1K2L7T10E4A80	52551884	31 000	180	6.74	38 547
11		2015	解放 CA1169PK2L2E4A80	60177107	15 800	121	4.04	35 744

1.4 预处理及 GC-MS 分析方法

典型烷烃预处理方法分为提取-浓缩-净化-再浓缩及定容这5个步骤。首先将样品(石英膜或PUF)加入二氯甲烷,超声提取3次,每次 30 min 。将提取液旋转蒸发至 1 mL 左右,加入填好 20 cm 硅胶和 10 cm 氧化铝的层析柱,用 50 mL 正己烷洗脱,收集洗脱液即为正构烷烃及藿烷类物质。最后将收集洗

脱液浓缩至 0.5 mL 左右,定容至 1 mL ,冷冻保存待测。

本研究分析仪器采用美国 Thermo 公司 Trace 1300-ISQ 气相色谱质谱联用仪,色谱柱为石英毛细管柱 DB-5MS ($30\text{ m}\times 0.25\text{ mm}\times 0.25\text{ }\mu\text{m}$, Agilent)。升温程序为初始温度 $50\text{ }^\circ\text{C}$,以 $6\text{ }^\circ\text{C}\cdot\text{min}^{-1}$ 升至 $300\text{ }^\circ\text{C}$,保持 15 min 。不分流进样, $1\text{ }\mu\text{L}$ 。进样口温度

为 275℃, 检测器温度 280℃, 传输线温度 280℃, 载气为高纯氮气(99.999%), 流速为 1.5 mL·min⁻¹, 溶剂延迟 5 min. 定量分析采用选择离子模式, 分别检测 $m/z = 85$ 正构烷烃, $m/z = 191$ 藿烷类.

采用外标法对典型烷烃进行定量分析, 定量物质包括 C15 ~ C35 21 种正构烷烃(美国

AccuStandard 公司, 99.9%), 17 α (H), 21 β (H)-C30 藿烷(C30-藿烷), 22R-17 α (H), 21 β (H)-C31 升藿烷(22R-C31 升藿烷)以及 22S-17 α (H), 21 β (H)-C31 升藿烷(22S-C31 升藿烷)(挪威 Chiron AS 公司, 99.9%), 其标准曲线、相关系数(R^2)及检出限如表 2 所示.

表 2 正构烷烃及藿烷类标准曲线、 R^2 及检出限

Table 2 Standard curves, R^2 , and the detecting limit of *n*-alkanes and hopanes

项目	物质	线性方程	R^2	检出限/ng·mL ⁻¹
正构烷烃	C15	$y = 27\ 874\ 921.25x + 1\ 574\ 791.30$	0.999 7	2.75
	C16	$y = 30\ 214\ 730.27x + 926\ 417.61$	0.999 8	2.67
	C17	$y = 33\ 222\ 664.05x + 1\ 271\ 943.23$	0.999 8	2.50
	C18	$y = 34\ 848\ 856.98x - 459\ 057.92$	1.000 0	2.59
	C19	$y = 38\ 175\ 446.23x + 806\ 495.74$	0.999 9	2.54
	C20	$y = 40\ 282\ 265.54x - 777\ 224.54$	0.999 9	2.52
	C21	$y = 42\ 007\ 783.4x - 563\ 875.54$	0.999 8	2.54
	C22	$y = 44\ 803\ 705.36x - 2\ 014\ 785.93$	0.999 5	2.66
	C23	$y = 45\ 987\ 424.94x - 2\ 258\ 783.59$	0.999 4	2.72
	C24	$y = 44\ 978\ 684.42x - 1\ 954\ 426.21$	0.999 6	2.89
	C25	$y = 44\ 289\ 534.42x - 1\ 883\ 354.73$	0.999 5	3.13
	C26	$y = 42\ 765\ 443.43x - 2\ 011\ 686.30$	0.999 3	3.10
	C27	$y = 41\ 632\ 628.97x - 3\ 192\ 144.29$	0.998 5	3.39
	C28	$y = 40\ 010\ 911.08x - 3\ 401\ 150.13$	0.998 2	3.52
	C29	$y = 38\ 155\ 989.22x - 3\ 842\ 866.61$	0.997 6	4.04
	C30	$y = 36\ 964\ 859.79x - 4\ 729\ 625.84$	0.996 1	4.33
	C31	$y = 35\ 908\ 508.64x - 5\ 454\ 822.15$	0.993 9	4.86
	C32	$y = 24\ 983\ 796.15x - 1\ 690\ 382.37$	0.994 2	5.77
	C33	$y = 21\ 373\ 399.18x - 1\ 511\ 327.94$	0.992 1	6.91
	C34	$y = 17\ 662\ 202.43x - 1\ 112\ 880.54$	0.994 5	8.93
	C35	$y = 14\ 339\ 971.18x - 1\ 054\ 785.21$	0.994 1	11.36
藿烷类有机物	22S-C31 升藿烷	$y = 2\ 321\ 745.88x - 24\ 894.25$	0.9998	3.47
	22R-C31 升藿烷	$y = 25\ 676\ 669.15x - 651\ 153.79$	0.998 6	2.93
	C30-藿烷	$y = 159\ 028\ 482.20x - 5\ 314\ 214.54$	0.997 6	4.65

1.5 质量保证措施

为保证测试具有较好的重复性, 本研究对所有测试车辆, 在道路行驶采样中进行一次相同路线下的重复采样. 并设置样品空白和溶剂空白, 上述空白实验均未检出待测污染物. 为保证分析仪器运行的稳定性, 本研究使用标准品连续进样 5 次, 计算 RSD 值, 其值小于 0.18%, 仪器运行良好.

2 结果与讨论

2.1 正构烷烃

2.1.1 排放标准对正构烷烃排放的影响

(1) 正构烷烃排放因子

如图 1 所示, 相对于国 I 测试车辆而言, 国 II 测试车辆排放因子有所增加, 国 III、国 IV 测试车辆排放因子明显下降. 国 IV 测试车辆正构烷烃气固相及总排放因子相比于国 I 测试车辆而言, 分别降低了

71.59%、73.35% 和 72.23%. 气相正构烷烃排放因子均高于固相排放因子, 平均是固相的 1.71 倍. Perrone 等^[9] 研究欧 I ~ 欧 IV 标准柴油车正构烷烃 C20 ~ C32 的排放因子, 也发现正构烷烃的排放与排放标准有很大关系. 欧 I 测试车辆正构烷烃排放因子为 2034 $\mu\text{g}\cdot\text{km}^{-1}$, 欧 III、欧 IV 测试车辆正构烷烃排放因子相对持平, 分别为 87 $\mu\text{g}\cdot\text{km}^{-1}$ 和 101 $\mu\text{g}\cdot\text{km}^{-1}$, 相比于欧 I 分别降低了 95.72% 和 95.03%. Liu 等^[19] 等研究也表明现代的发动机和技术标准能够显著降低碳氢化合物排放. 本研究正构烷烃排放因子要明显高于 Perrone 等研究, 主要原因是测试车型不同, 且检测的碳数范围小于本研究, 各国不同排放标准、燃油种类以及测试车辆均会影响正构烷烃的排放^[20].

(2) 正构烷烃碳数分布及特征参数

如图 2 所示, 整体上气相正构烷烃呈双峰前锋

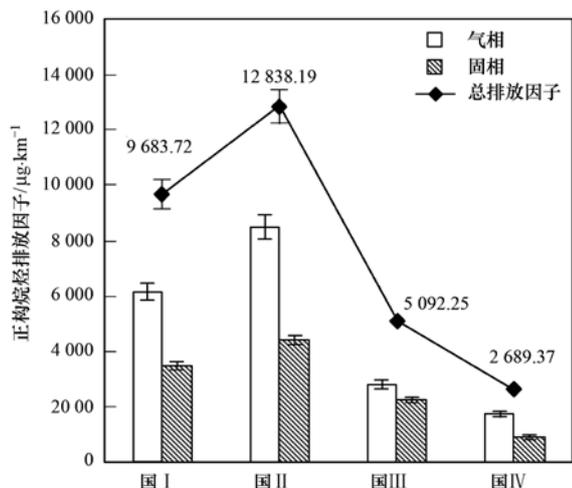


图1 国 I ~ 国 IV 标准测试车辆正构烷烃气固相及总排放因子
Fig. 1 Gaseous, particulate phase and total emission factors of *n*-alkanes from China I to China IV test vehicles

型,以 C17 ~ C18 为主峰碳;固相呈单峰前锋型,以 C18 ~ C21 为主峰碳,说明固相相对于气相而言,更易富集高分子量的烷烃. 研究表明^[21] 主峰碳数可作为有机质来源和成熟度的标志,人为使用的化石燃料一般成熟度较高,主峰碳数 ≤ 23 ,且无明显的奇偶优势^[22]. 上述气固相正构烷烃主峰碳数均小于 C23,符合化石燃料源输入的特征. 李登科等^[4]采用台架测试研究国 II、国 III 柴油发动机也显示气态正构烷烃以 C15 ~ C17 为主峰碳,固相正构烷烃主峰碳为 C19 ~ C22. 除此之外,国 III、国 IV 测试车辆气固相正构烷烃在 C27 ~ C31 之间均显示“锯齿”状,有一定的奇偶优势,并且国 I 气相和国 III 固相正构烷烃以 C26 ~ C27 为主峰碳. 分析其原因可能是由于在采样稀释尾气过程中,稀释空气存在少量的植

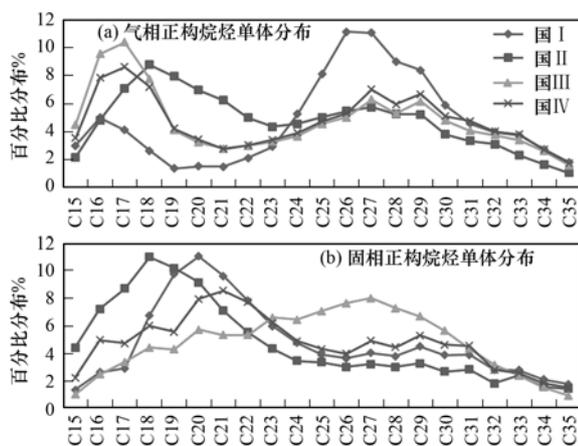


图2 国 I 至国 IV 标准测试车辆正构烷烃气固相碳数百分比分布特征

Fig. 2 Percentage contribution of each *n*-alkane in the emissions of China I to China IV test vehicles

物碎屑,使采集尾气混入了生物源^[23].

由表 3 可以看出,4 种标准测试车辆气固相正构烷烃 CPI 值变化范围在 1.01 ~ 1.06 之间,平均值为 1.03 非常接近于 1; % waxC_n 植物蜡贡献率在 1.67% ~ 6.65% 之间,平均值为 3.90%. 本研究中 L/H 的值,其变化范围在 0.42 ~ 2.47 之间,除个别值外,大多大于或接近于 1,符合石油输入的特征^[24].

表 3 国 I 至国 IV 标准测试车辆正构烷烃特征参数

Table 3 Characteristic parameters of *n*-alkanes emitted from China I to China IV test vehicles

技术	项目	主峰碳	CPI	Wax%	L/H
国 I	气相	C26	1.03	4.39	0.42
	固相	C20	1.03	3.24	1.68
国 II	气相	C18	1.02	1.78	1.38
	固相	C18	1.06	2.56	2.47
国 III	气相	C17	1.05	1.67	1.10
	固相	C27	1.01	6.65	0.82
国 IV	气相	C17	1.06	6.61	0.93
	固相	C21	1.03	4.34	1.44

2.1.2 行驶工况对正构烷烃排放的影响

(1) 正构烷烃排放因子及特征参数

如图 3 所示,工况对正构烷烃的排放有明显影响. 高速工况下,测试车辆正构烷烃平均气固相及总排放因子分别为 2 579.90、2 132.40 和 4 712.30 $\mu\text{g}\cdot\text{km}^{-1}$;而非高速工况下为 6 241.26、3 604.36 和 9 845.62 $\mu\text{g}\cdot\text{km}^{-1}$;非高速工况下测试车辆平均气固相及总排放因子分别是高速工况的 2.42 倍、1.69 倍和 2.09 倍. 关共凑^[25]研究佛山市区机动车尾气污染排放特征时也发现,慢速或怠速工况下机动车

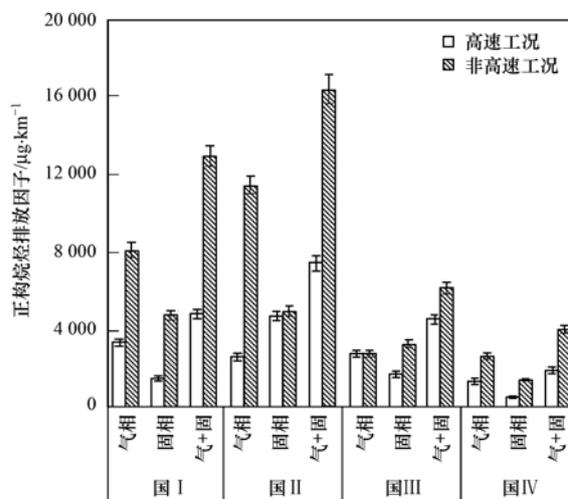


图3 不同行驶工况测试车辆正构烷烃气固相及总排放因子

Fig. 3 Gaseous, particulate phase and total emission factors of *n*-alkanes in different driving conditions

尾气会产生更多污染物,排放因子更高. 测试车辆

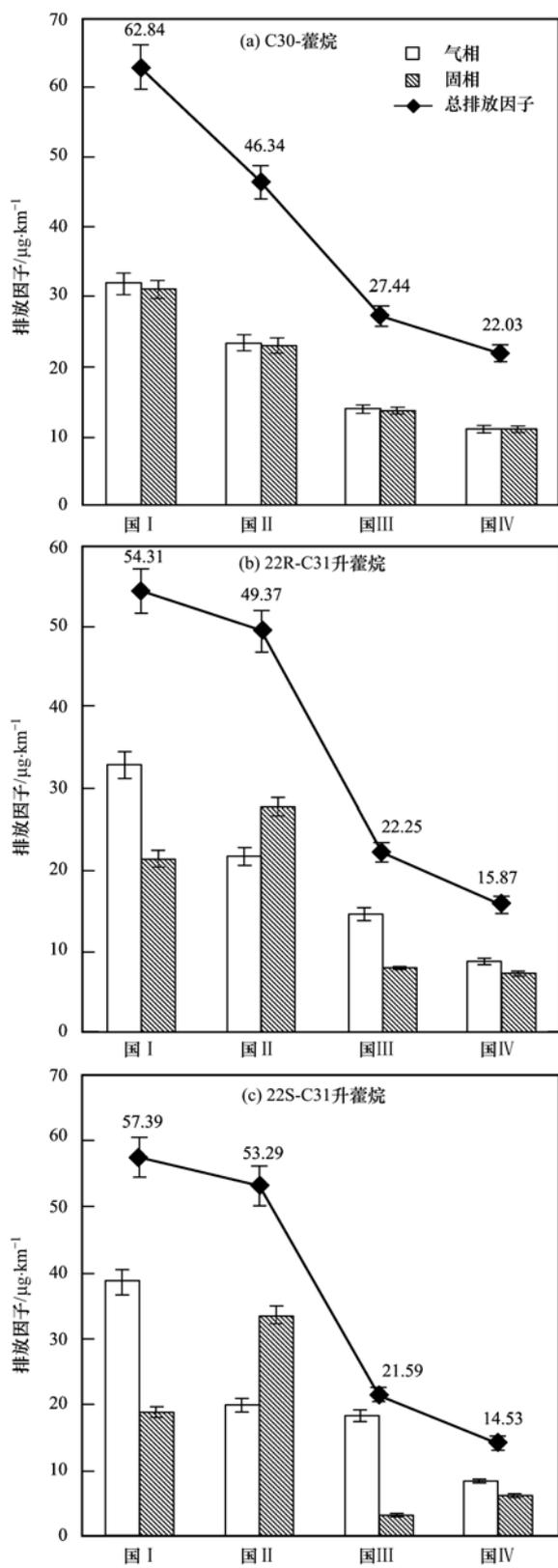


图 4 国 I 至国 IV 标准测试车辆藿烷类有机物气固相及总排放因子

Fig. 4 Gaseous, particulate phase, and total emission factors of hopanes emitted from China I to China IV test vehicles

在高速工况和非高速工况下正构烷烃碳数分布和主要特征参数没有明显的差异规律,其结果与排放标准对正构烷烃排放影响中讨论结果相似.

2.2 藿烷类有机物

2.2.1 排放标准对藿烷类有机物排放特征影响

(1) 藿烷类有机物排放因子

如图 4(a) 所示,测试车辆 C30-藿烷总排放因子由国 I 测试车辆($62.84 \mu\text{g}\cdot\text{km}^{-1}$)降低至国 IV 测试车辆($22.03 \mu\text{g}\cdot\text{km}^{-1}$),降幅达到 64.95%;且 C30-藿烷气固两相排放因子非常相近. 由图 4(b) 和图 4(c) 可以看出,22R-C31 升藿烷和 22S-C31 升藿烷排放因子和排放特征大致相同. 相比于国 I 而言,国 IV 测试车辆总排放因子分别降低了 70.78% 和 74.68%. 二者排放因子总体上均显示气相大于固相,平均是固相的 2.39 倍和 1.36 倍. 4 种标准测试车辆 C31 升藿烷 22S/(22S + 22R) 的比值没有较大差异,范围在 0.46 ~ 0.56 之间,平均值为 0.50,已接近平衡终点 [$S/(S + R) = 0.60$ 左右],与成熟度较高的石油中藿烷类有机物分布特征十分接近^[26].

(2) C30-藿烷相关性分析

如图 5 所示,正构烷烃总排放因子与 C30-藿烷总排放因子呈现明显线性关系,其 R^2 为 0.9268. Huang 等^[27] 研究柴油机半挥发性有机物排放特征时,发现 $\sum 15\text{PAHs}$ 、 $\sum 11\text{NPAHs}$ 、 $\text{PM}_{2.5}$ 以及 SOF 与 C30-藿烷排放有显著的线性相关性,其线性相关系数 R^2 在 0.72 ~ 0.95 之间. 但已有研究中 C30-藿烷与各污染物比值仍存在很大波动范围与差距. Phuleria 等^[28] 研究 $\sum 9\text{PAHs}$ 与 C30-藿烷比值发现,其波动范围在 0.6 ~ 5400 之间. 本研究通过计

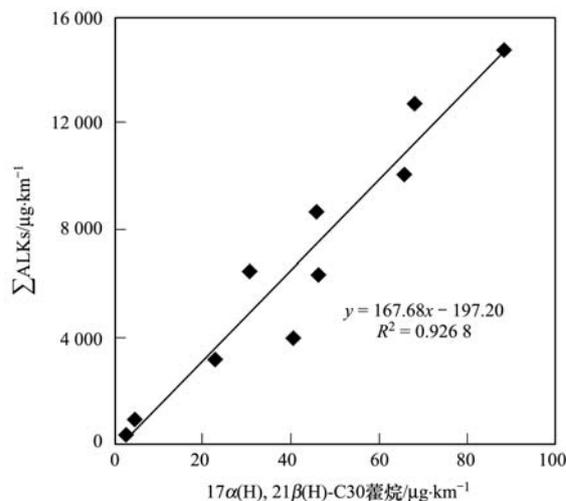


图 5 11 辆测试车辆正构烷烃与 C30-藿烷相关性分析

Fig. 5 Linear relationship of total *n*-alkanes and C30hopanes

算得出了正构烷烃与 C30-藿烷的线性关系及相关

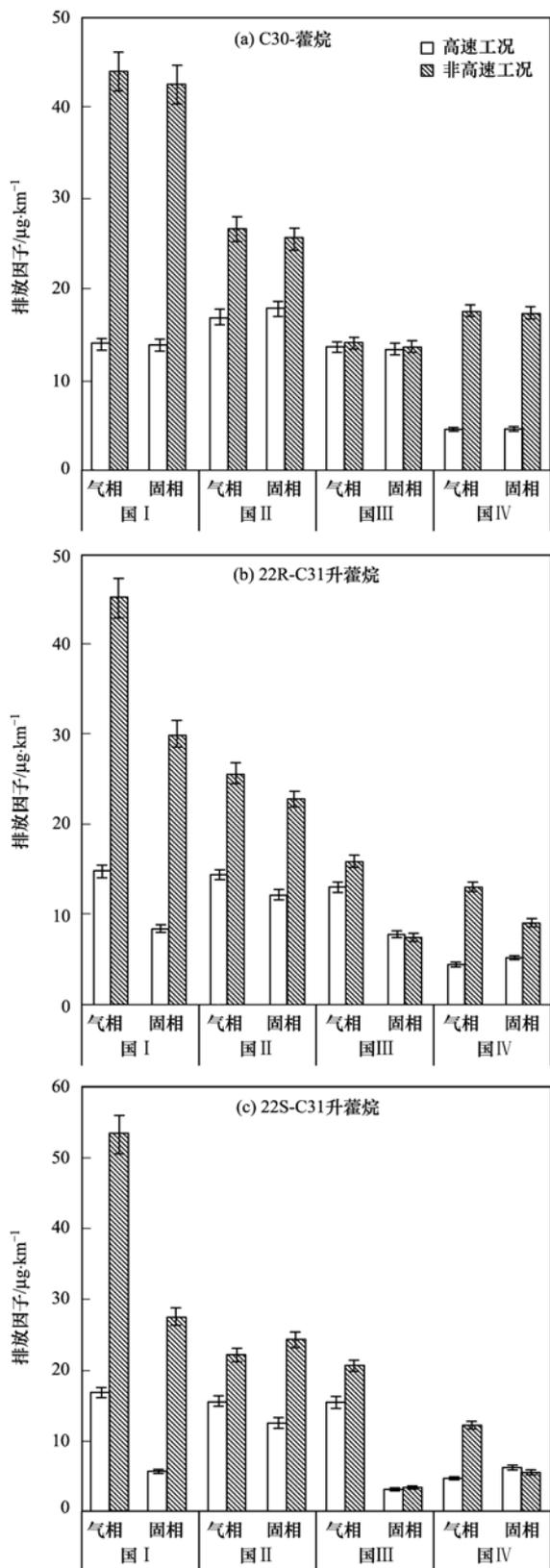


图 6 不同行驶工况测试车藿烷类有机物气固相及总排放因子

Fig. 6 Gaseous, particulate phase, and total emission factors of hopanes in different driving conditions

规律, 希望能够为 C30-藿烷相关性分析提供一定理论及数据支持, 为今后使用 C30-藿烷来定量示踪机动车源解析方法奠定基础.

2.2.2 不同工况藿烷类有机物排放因子及相关性分析

图 6 给出了测试车辆在高速工况和非高速工况下藿烷类有机物气固相及总排放因子. 在高速工况下, C30-藿烷, 22R-C31 升藿烷和 22S-C31 升藿烷平均总排放因子分别为 24.70、20.07 和 20.05 $\mu\text{g}\cdot\text{km}^{-1}$; 而非高速工况下, 三者平均总排放因子分别为 50.22、42.18 和 42.20 $\mu\text{g}\cdot\text{km}^{-1}$. 测试车辆在非高速工况下 3 种藿烷类有机物平均总排放因子是高速工况的 2.03 ~ 2.10 倍. 高速工况和非高速工况下测试车辆 22S/(22S + 22R) 比值没有较大差别, 范围在 0.38 ~ 0.56 之间, 平均值为 0.48. 正构烷烃与 C30-藿烷也有较好的相关性, R^2 分别为 0.978 1 和 0.803 6.

3 结论

(1) 排放标准对测试车辆正构烷烃、藿烷类有机物的排放均有显著影响. 整体上排放因子随标准的提升而降低, 其中国 III、国 IV 测试车辆排放因子降低显著.

(2) 测试车辆在高速工况和非高速工况下正构烷烃、藿烷类有机物的排放也有显著差异, 非高速工况下是高速工况的 1.69 ~ 2.42 倍.

(3) 11 辆测试车辆正构烷烃总排放因子与 C30-藿烷总排放因子在不同排放标准及行驶工况下均呈现出一定的线性关系, 其 R^2 范围在 0.803 6 ~ 0.978 1 之间.

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