甲醇燃料车尾气净化催化剂的研究(I)

——多组分催化剂对甲醇的深度氧化

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摘要 利用色谱-微反联用技术研究多种多元催化剂对甲醇深度氧化的活性,并借助于 XRDBET 等手段考察焙烧温度对活性的影响。结果表明,不同的催化剂对甲醇氧化时均有甲醛及甲酸甲酯伴生,其伴生温度区间及最大伴生浓度随不同催化剂而变化;32-37 号催化剂的活性明显高于双组分催化剂;添加少量稀土金属氧化物 CeO₂ 和贵金属 Pd 对活性均有一定的改善作用;提高焙烧温度,催化剂表面发生烧结,对活性有不良影响。 关键词 甲醇,甲醛,甲酸甲酯,深度氧化,催化剂,甲醇燃料。

目前甲醇燃料作为汽油代用品已经在国外 市场上大量销售^[1]。尽管甲醇燃料被誉为"清洁 燃料",但是,其燃烧尾气中仍含有相当成分的污 染物,如未完全燃烧的甲醇以及部分氧化产物甲 醛及甲酸甲酯等^[2],这些都给汽车尾气净化的研 究带来了新课题。近几年来,本所致力于开发新 型催化剂以适应市场的迫切需要,取得了一定进 展。本文报道有关二元,三元以及多元组分非贵 金属氧化物催化剂对甲醇的深度氧化情况。

1 实验部分

1.1 催化剂制备

将一定量的催化剂活性组分前体物质如硝酸盐按要求配成溶液,用 20—40 目载体 r-Al₂O₃ (比表面积 195m²/g,孔容为 0.76ml/g)浸渍该溶 液,在 110℃干燥 5h,在 200℃焙烧 2h,升温至 500℃焙烧 3h,制成成品催化剂。其中 Ce-O 是利 用 Ce(NO₃)₃ • 6H₂O 溶液在已制备好的催化剂 上再次浸渍,采用同样的焙烧条件负载的。

1.2 催化剂的活性评价

采用色谱-微反联用技术,催化剂填充量为 0.5g,空速 12000h⁻¹,甲醇浓度 3000×10⁻⁶,氧 的浓度 5%。尾气分析采用沪产 GC-102 色谱仪 氢焰离子检测器测定,规定甲醇等转化率为 50%,95%时对应的床层温度为 T_{50} , T_{95} ,甲醇氧 化生成甲醛、甲酸甲酯的初始温度为 $T_{\pm\alpha}$,甲醛 及甲酸甲酯消失时温度为 T_{n+} ,最大浓度对应的 温度为 $T_{\text{wft}}(C)$;该最大浓度为 $c_{\text{wft}}(10^{-6})$ 。

1.3 X-射线衍射分析(XRD)

采用日本松下公司 D/max Ⅱ型衍射仪,以 Cu Ka 为辐射线源,工作条件为 40kV×40mA。

1.4 催化剂比表面测定(BET)

测定仪是 ST-03 型,称样为 0.2g,在 120℃ 干燥 2h,采用常压流动法在液氛温度下吸附,在 室温下脱附。采用 BET 方程计算表面积。

2 结果与讨论

2.1 二元组分催化剂对甲醇的氧化活性

单组分催化剂的研究表明: $Cu-O/r-Al_2O_3$, Co-O/r-Al_2O_3 以及 Ag-O/r-Al_2O_3 是优良的甲醇 深度氧化催化剂^[3]; Mn-O 中 Mn 元素易变价并 且该物质传递氧的性能较好^[4],故选择以上 4 种 物质作为二元组分催化剂的活性组分。表 1 是 Cu-Co-O/r-Al_2O_3, Cu-Mn-O/r-Al_2O_3 以及 Cu-Ag-O/r-Al_2O_3 等多种催化剂的活性测试结果。由此 可知: 当 Cu: Co=3:1(原子比)时,催化剂的活 性较好; Cu: Mn=1:1(原子比)时活性也较优; 而 Cu-Ag-O 中,随 Ag 的含量渐次提高,其催化 剂的活性相应地提高,双元组分的 Cu-Ag-O/r-Al_2O_3 比 Cu-O/r-Al_2O_3 活性好,但仍逊于 Ag-O/

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表 1

不同催化剂的活性比较

序 号

序 号 催化剂 组成 Cu-O Cu-Co-O Cu-Co-O Cu-Co-O Co-0 Cu-Mn-O Cu-Mn-O Cu-Ag-O Cu-Ag-O Cu-Ag-O 原子比 3:11:1 1:3 3:1 1:1 1:3 20:1 15:1 10:1 甲醇 T_{50} T_{95} 甲醛 $T_{\pm id}$ T峰宙 T消失 C峰值 甲酸甲酯 T生成 T_{iff} T消失 ℃峰值

r-Al₂O₃,但考虑到 Ag 是贵重金属,其含量不宜 太高,一般应低于总活性组分的5%,由于含量 太低的单组分 Ag-O/r-Al₂O₃ 氧化活性会降低, 因此采用多组分催化剂是比较合适的。表1数据 说明:副产物甲醛及甲酸甲酯的伴生浓度与催化 剂组成也有一定联系。其中活性较好的 Cu-Co-O/r-Al₂O₃、Cu-Co-O/r-Al₂O₃ 副产物伴生浓度相 应较低;Cu-Ag-O/r-Al₂O₃的氧化活性尽管没有 Ag-O/r-Al2O3 活性高[3],但其副产物伴生浓度却

Mn 的含量

依次增加

Cu:Ag不变,但

小于 32 号; Mn

的含量依次升高

明显地低于后者,甲醛和甲酸甲酯的最大伴生浓 度仅为 Ag-O/r-Al₂O₃ 的 48%和 47%。

三元组分催化剂对甲醇的氧化活性 2.2

32—37 号催化剂是 Cu、Mn、Ag 按照一定的 配比制成的 3 元组分催化剂,其中 32-34 号催 化剂中 Cu: Ag 的原子比保持不变, Mn 的含量 依次增加;35—37 号催化剂中 Cu: Ag 原子比不 变但小干 32 号, Mn 的含量依次增加。表 2 是这 6种催化剂的活性测试结果。

T消失

C峰值

< 50

< 50

 催化剂组成	甲	醇		甲	醛			甲酸	甲酯
Cu : Ag : Mn	T ₅₀	T ₉₅	T _{生成}	T _{蜂ቢ}	T _{消失}	C峰值	T _{生成}	T _{峰值}	7
Cu:Ag 不变	150	195	140	155	315	80	110	130	21

表 2 3 元组分催化剂的活性比较

表2结果表明:①Cu-Ag-Mn-O/r-Al₂O₃对 甲醇的氧化活性比2元组分有明显提高;②催化 剂的活性随 Mn、Ag 的含量增加而提高。其中 37 号催化剂的 T₅₀、T₉₅低达 120℃和 145℃。该催化 剂中 Cu: Ag: Mn 的比例基本上与 2 元催化剂 Cu:Ag、Cu:Mn 相一致。由此可知3者的合理 匹配可以产生良好的协同效应。③甲醛和甲酸甲 酯的伴生浓度明显地低于2元催化剂。37号催 化剂上,2者的最大伴生浓度均小于 50×10-6。 2.3 添加稀土氧化物 CeO₂ 对活性的影响

< 50

< 50

< 50

< 50

添加 Ce 的目的是希望改善 Cu-Ag-Mn-O/r-Al₂O₃催化剂的贮氧性能,以便达到提高活性的 目的。以 37 号 Cu-Ag-Mn-O/r-Al₂O₃ 催化剂的配 比为基础 配方,再分别浸渍 2%、5%、8%的 CeO2,制成 38-39A 3 个催化剂。考虑到 CeO2 对 甲醇的氧化活性较差,含量太高可能不利于提高 活性,因此未进一步对更高含铈的催化剂加以研究。实验结果如表3所示。结果表明:加入铈之后,催化剂的活性与未加入时相比有一定的提高,38号与37号相比,甲醇氧化的T₅₀,T₉₅分别

降低了 5℃,尽管幅度不大,但对高活性催化剂, 其活性进一步增加比较困难。铈的含量以 5%为 宜。

分别 2.4 添加贵金属 Pd 的影响

催化剂			甲	醇		甲	醛	Y 甲酸甲酯					
序	:号	组	成	T 50	T 95	T _{生成}	T _{峰值}	T _{消失}	C峰值	T生成	T峰值	T _{消失}	C峰值
38	Cu-	Ag-Mn+	2%Ce	120	140	105	140	300	<50	100	120	180	<50
39	Cu-	Ag-Mn+	5%Ce	120	140	100	140	295	<50	95	110	180	<50
39A	Cu-	Ag-Mn+	8%Ce	120	145	110	140	300	<50	90	110	180	<50

表 3 添加稀土铈对催化剂活性的影响

把微量的金属钯引入 37 号 Cu-Mn-Ag-O/r-Al₂O₃ 和 39 号的 Cu-Ag-Mn-Ce-O/r-Al₂O₃ 催化剂 之中,制得 40—43 4 种催化剂,其中 Pd 的含量 依次增加,实验结果列入表 4。由表 4 中数据可 知:引入 Pd 之后催化剂的活性有一定的变化。 40—41 号催化剂的活性有一定提高。但随 Pd 的 相对值增加,42、43 号 2 个催化剂活性反而降 低,而且甲醛及甲酸甲酯的伴生浓度也增加。已 知 Pd-O 的甲醇氧化活性较低,由于加入 Pd 之后 主要是以 Pd-O 的形式存在而未加还原处理,低 活性 Pd-O 占据了一定的表面活性位,以致于高 活性的表面分率下降,造成了总活性下降,故 Pd 的含量不宜过高。

2.5 焙烧温度对催化剂活性的影响

表 4 添加贵金属 Pd 对催化剂活性的影响

庐 — —	催任	上剂组质	戉	甲	醇		甲	醛		甲酸甲酯				
17 5	Cu : Ag : Mn	Ce %	Pd 的相对值	T 50	T ₉₅	T _{生成}	T峰值	T _{消失}	C峰值	T生成	T _{峰值}	T _{消失}	 C峰值	
39	同	5		120	140	110	140	295	<50	95	110	180	<50	
40	37		1	120	145	105	140	290	<50	95	110	180	<50	
41	号	5	1	120	135	100	130	290	<50	90	105	175	<50	
42	_	5	5	130	170	95	135	310	120	90	110	180	100	
43	样	5	10	165	1 85	95	140	340	250	90	120	180	180	



图 1 甲醇转化率、催化剂比表面与焙烧温度的关系

取 37 号催化剂在空气和 600℃、700℃、 800℃、900℃、1000℃条件下分别焙烧 4h,然后 在相同条件下进行活性评价。结果如图 1 所示。 可以看出,甲醇转化率随催化剂的焙烧温度的提 高而呈下降趋势。由此表面数据可知,随温度升



高,催化剂的比表面积下降,活性下降与比表面 积下降有明显的对应关系。除此之外,高温引起 催化剂化学结构的变化也可能是活性下降的原因。图 2 为不同温度下的 XRD 谱图,随温度增加,催化剂晶粒发生变化,结晶度下降,1000℃时呈弥散状态,说明催化剂表面活性组分发生了烧结,这与比表面数据相一致。

 6 不同催化剂对应的副产物伴生温度区间及 峰值浓度曲线

在甲醇氧化时,几乎所有催化剂上均产生一 定量的甲醛及甲酸甲酯,但不同的催化剂,二者 的生成温度和消失温度不同,其峰值浓度也各 异。笔者将本研究所涉及的单组分,双组分,3组 分以及多组分催化剂按序号自左至右排列,把甲 醇氧化时甲醛及甲酸甲酯的生成温度,消失温 度,峰值温度以及峰值浓度对催化剂序号绘图 (图 3-4)。由于催化剂的活性基本上符合由单 组分、双组分到多组分的递增顺序,故可以近似 地认为,催化剂的序号顺序就代表了活性递增顺 序。称不同催化剂的甲醛及甲酸甲酯的生成温度 和消失温度所联成的曲线为生成温度曲线和消 失温度曲线,二者之间的区域为伴生区间。





由图 3 和图 4 可以得出如下重要结果:

(1)从峰值温度曲线上看,自左至右随催化 剂活性的增加向低温方向移动,其中 14 号催化 剂 Ce-O/r-Al₂O₃ 甲醛和甲酸甲酯峰值温度高达 290℃和 295℃,而 40 号 Cu-Ag-Mn-Pd-O/r-Al₂O₃ 只有 140℃和 110℃。 (2)从峰值浓度曲线来看,总趋势也是自左 至右呈下降趋势。其中14号 Ce-O/r-Al₂O₃,16号 Pd-O/r-Al₂O₃和18号 Pd/r-Al₂O₃3种催化剂产 生的甲醛、甲酸甲酯浓度明显较高。但36—41号 多元组分催化剂,2者的伴生浓度都低于50× 10⁻⁶,说明活性越高,副产物的(下转第60页)

(5)可以进行一条公路上有不同源强路段情

理;

特点;

(3)没有复杂的积分计算;

(4)虚拟线源比虚拟点源更接近实际线源的 况下的计算;

时间	计算与实测值	10m	50m	100m	150m	200m	R	S_{XY}/\overline{X}
1988-12-24	HIGHWAY- I 计算值	4.92	1.76	1.27	1.05	0.096	0.88	0.30
	计算值	3.97	2.13	1.86	1.15	1.03	0.85	0.25
	实测值	3.81	1.82	1.94	0.09	2.15		
1989-07-19	HIGHWAY- I 计算值	3.74	0.97	0.54	0.39	0.03	0.99	1.55
	计算值	4.37	2.26	1.09	0.58	0.21	0.98	0.62
	实测值	6.90	2.40	1.30	0.12			

表 2 京顺公路示踪剂浓度实测值与计算值(mg/m³)

表 3 沪嘉高速公路 CO 分布计算值与实测值(mg/m³)

样本号	计算与实测值	ر 15m	25m	55m	75m	95m	R	S_{XY}/\overline{X}
116	CLINE2 计算值	1.94	1.97	1.98	1.97	1.96	0.87	0.62
(45°)	计算值	1.65	2.62	3.25	1.86	1.75	0.89	0.11
	实测值	1.30	2.90	3.20	1.53	2.35		
117	CLINE2 计算值	0.051	0.085	0. 086	0.075	0.069	无相关性	
(0°)	计算值	0.95	0.83	0.70	0.69	0.59	0.95	0.022
	实测值	0.87	0.77	0.74	0.59	0.69		
118	CLINE2 计算值	1.91	1.96	1.98	1.97	1.96	0.51	1.64
(90°)	计算值	1.93	3.02	3.58	2.03	1.91	0.82	0.21
	实测值	1.71	3.93	3.99	1.95	1.87		
210	CLINE2 计算值	1.60	1.69	1.69	1.68	1.66	0.88	0.18
(67°)	计算值	1.35	1.42	1.52	1.79	1.63	0.85	0.25
	实测值	2.46	1.40	1.49	1.86	1.65		

(6)对路堑的情况,仍可按本方法计算,但平面上 初期扩散混合区的源强与实际源强有一定的稀 释度。

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(上接第37页)浓度越低。

(3)甲酸甲酯的生成温度曲线和消失温度曲 线出现了明显变化,Ce-O/r-Al₂O₃ 的 2 值较大, 故出现了 1 个峰,但总趋势自左至右渐次降低, 32 号催化剂以后的两温度曲线都分别低于 130℃和 180℃。而且 2 者之间的距离越来越接 近,最窄时只有 70℃左右。故甲酸甲酯的伴生区 间随催化剂活性增加而变窄。

(4)对于 32—41 号催化剂对应的甲醛及甲 酸甲酯的峰值温度曲线和生成温度曲线,发现 2 者靠的很近,大约相差只有 20—30℃左右,并且 远离消失温度曲线,这说明 32—41 号催化剂在 对甲醇氧化时,在一个比较窄的温度区间内,甲 醛和甲酸甲酯的伴生浓度达到最大,然后迅速降 低到 50×10⁻⁶以下,如果把 50×10⁻⁶以下的甲 酸甲酯或甲醛完全氧化,则需要更高的温度。

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Abstracts

Chinese Journal of Environmental Science

a waterborne salt movement was found to be 120 cm water column pressure. However, a 30 cm thick clay layer artificially covered on the top surface of the original sediment shows a strong suppression against the waterborne salt movement even if the suction head is over 800 cm water column prissure. No salt content was found to be accumulated on the surface during a 3 month period of simulated evaporation.

Key words: oxidative pond sediment, waterborne salt transport, simulation.

Application of GKS in National Water Quality Management Information System. Jiang Yong, Fu Guowei (Department of Environmental Engineering, Tsinghua University, Beijing 100084): Chin. J. Environ. Sci., 15(3), 1994, pp. 22-25

GKS (Graphics Kernel System), the first ISO international standard for computer graphics, was used to construct a graphic application system with a higher efficiency of development and a stronger implantability of programs. This paper also deals briefly with the position of GKS in various applications, major functions and the principles of developing such a graphic system. The reasonable functions of graphics were arranged to closely meet the requirements of the national information system water quality management. The software on structures of the graphic system, and the schemes of designing a general graphics and a geographic graphics, were also described along with a general evaluation on the effectiveness of GKS application in developing graphic systems.

Key words: water quality management, graphics kernal system, National Water Quality Management Information System.

Research of the Separation and Recovery of PER Plastic Wastes. Zhang Zhongyan, Zhao Genmei et al. (Department of Chemistry and Chemical Engineering, Shanghai University of Technology, Shanghai 200072); Chin. J. Environ. Sci., 15(3), 1994, pp. 26-29

The recycles of PET and high density polyethylene (HDPE) from the used plastic drinking bottles made of them were carried out by using a process comprising steps of: smashing the used bottles into pieces, air seperation, cleaning, and floating. With this process, PET, HDPE and other plastic wastes were effectively seperated and recovered at a rate of 97% for PET and a rate of 95% for HDPE. The recovered PET had a purity of above 95% and could be recycled as a raw material of plastics having a high quality.

Key words: PET, HDPE, separation and revovery of plastic wastes.

Use of Polymer Composite Materials for Vibration Damping and Noise Reduction. Zou Zongbai, Li Jun et al. (Southeast University, Nanjing 210018): *Chin. J. Environ. Sci.*, 15(3), 1994, pp. 30-33

A polymer composite material has been prepared by using an epoxy resin as binder, a low molecular weight polyamide as curing agent, and a mixture of powdered stone, quartz sand and glass fiber as filler, based on an optimized formulation designed to meet the requirements for the performance of material. The tests show that the composite material has a hardness of about 240 HB and an impact resistance of about $30 \text{kg} \cdot \text{cm}/\text{cm}^2$, both of which are close to those for some metal materials. This material is also easy to shape and is cost-effective. All of these make the material having a value of wide application, particularly use as alternatives to replace those metal parts which may undergo a heavier impact on shuttled textile machines in textile factories, in order to minimize the vibration and noise.

Key words: polymer composite material, epoxy resin, vibration damping, noise reduction.

Method for Treating Exhaust Gases from Methanol Fueled Internal Combustion Engine (I): A Deep Oxidation of Methanol over Multicomponent Catalysts. Wang Jin' an, Wang Ren (Institute of Industrial Catalysts, East China University of Science and Technology, Shanghai 200237): Chin. J. Environ. Sci., 15(3), 1994, pp. 34-37

Several kinds of multicomponent catalysts for a deep oxidation of methanol were studied for their activities and surface features by means of GC-MR, XRD, SEM, BET etc. The studies were also conducted on the effects of calcination temperature, oxygen level in the atmosphere and space-velocity on the activities of catalysts. The results show that the oxidation of methanol over all the catalysts studied produced formaldehyde (HCHO) and methyl formate $(HCOOCH_3)$, and both the temperature ranges in which they could be produced and the maximum concentrations at which they could be produced varied for different catalysts. Catalysts No. 32-37 were found to have a significantly high activity than the bicompont catalysts. The addition of a rare earth metal oxide (CeO_2) and a noble metal (Pd) in a small amount to such catalysts could improve their activities to an extent. Increased calcination temperatures would cause the catalyst surfaces to be sintered, making the activities reduced. Increased space- velocities could give a slightly higher rate of methanol convertion. The oxygen level was found to be preferably at 5% and a level of over 10% would have only a small effect on methanol convertion.

Key words; methanol, formaldehyde, methyl

Abstracts

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formate, deep oxidation, catalyst.

Study on the Recovery of Carbon Monoxide (CO) from Industrial Exhaust Gases by a Chemical Absorption Method. Su Chunhui, Che Yinchang et al. (Dept. of Nonferrous Metal., Northeastern University, Shen' yang 110006): Chin. J. Environ. Sci., 15(3), 1994, pp. 38-41

An aqueous CuCl-MgCl₂ system has been found to be a preferred, highly selective CO absorbent. A relationship between the maximum capacity of the absorbent to absorb CO and temperature was determined. The effects of a change in exhaust gas composition on CO recovery was also studied. The CO recovery with this absorption process was found to be up to 93%, and the recovered CO has a purity of 98% as determined by gas chromatography (GC). The CO gas can be desorbed from the CO absorbed absorbent liquor at temperatures in the range of 120-140 C. In addition, the mechanism of the absorption reaction between CO and the aqueous CuCl-MgCl₂ absorbent system was preliminarily studied. The new process can be used to separate and recover CO gas from industrial exhaust gases, such as the off-gas from steelmaking converters.

Key words: carbon monoxide, absorbent, chemical absorption

Atmospheric Dispersion Parameters for High **Overhead Pollution Sources Fitted** with the Monitored Data from Various Parts of China. Gu Yongrui, Zhang Tong, Wang Dongpu (Inner Mongolian Central Monitoring Station for Environmental Protection, Huhehaote 010010). Chin. J. Environ. Sci., 15(3), 1994, pp. 42-46

In a calculation of the atmospheric dispersion of emissions from a high overhead source, the atmospheric dispersion parameters given in the National Standards GB3840-91 and the Briggs parameters were found to be no longer suitable. By using the general expression for the Briggs atmospheric dispersion parameters ($\sigma = \alpha x (1 + \beta x)^{-1/2}$) to fit the atmospheric dispersion parameters which were actually measured in various parts of China, the fitted parameters were obtained and were found more reasonable as compared with the GB3840-91 and Briggs parameters, and thus have a practical value in use for calculating other atmospheric dispersion parameters.

Key words: atmospheric dispersion, fitting, high overhead sources.

Determination of the Source Intensity of the Gases Released from Municipal Solid Wastes Dumping sites and Their Environmental Impact Assessment. Zhou Zhongping, Zhang Jun (Dept. of Environ. Eng., Tsinghua University, Beijing 100084): Chin. J. Environ. Sci., 15(3), 1994, pp. 47-52

After taking samples of the gases released from the Beishenshu Municipal Solid Wastes Dumping Site in Beijing and making the qualtative and quantitative analyses of the samples, two methods were used to study the determination of the intensity of the gases releasing sources, by which an assessment was made on the environmental impact of the gases emitted from the dumped garbage. Some countermeasures feasible to control such a pollution were suggested.

Key words: gases release, garbage dumping site, source intensity, EIA.

Forms and Transformation of Chromium Species in Soils. Chen Yingxu, He Zeng' yao et al. (Dept. of Environmental Protection, Zhejiang University of Agriculture, Hangzhou 310029); Chin. J. Environ. Sci., 15(3), 1994, pp. 53-56

developing a method for the fractional extraction of chromium species in various binding states in soil, it was found that the extractants of 1 $mol/L NH_4Ac$, 2 mol/L HCl and 5% H_2O_2 -2mol/L HCl in use for a sequential extraction of chromium species from soil can give the exchangeable Cr species, precipitated Cr species, and organics-bound Cr species, respectively. The results show that in the natural soil the Cr speies are present dominantly in a precipitated or residual state. Under the reducing conditions, the Cr species in soil tend to be transformed into those in an organics-bound state. As the soil pH value was lowered, the levels of water soluble Cr species and exchangeable Cr species raised while the levels of Cr species in precipitated or residual state being reduced. The soil pH value can be lowered by adding Cr(II) species and raised by adding Cr (VI) species.

Key words: chromium, soil, fractional extraction, species transformation.

Mathematical Modelling on the Dispersion of Line Sources of Air Pollution. Cheng Zirun, Fu Dafang (Institute of Environmental Engineering, Southeast University, Nanjing 210018); Chin. J. Environ. Sci., 15(3), 1994, pp. 57-60

Based on the traditional Gaussian dispersion theory, a method has been proposed to calculate the dispersion of line sources of air pollution caused by vehicles runing on road. In this method, a road line source is divided into several elements in which an initial dispersion exists; each of the elements is considered to be a proximate short line source which is passing through the midpoint of the element and is rectangular to the direction of wind, and can be calculated for its dispersion based on the Gaussian Model for rectangular wing, with the concentration