# 高浓度难降解有机废水低压湿式催化氧化处理*

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摘要 在湿式空气氧化法和Fenton试剂的基础上, 研究了一种新的低压湿式催化氧化法. 该法与湿式空气氧化法相比, 压力为0.1–0.6MPa, 而后者压力为3.5–10MPa; 温度小于180℃; 与Fenton法相比, 当$H_2O_2$·COD(重量比)小于1.2时, 对COD大于14000mg/L的含酚废水, COD去除率提高20%以上, 试验证实硫酸在加温、加压(0.1–0.6MPa, 104–165℃)条件下对Fenton试剂除COD具有协同作用. 用该法还进行了部分染料和农药废水处理研究.

关键词 湿式氧化, Fenton试剂, 催化氧化, 有机废水, 废水处理.

由美国Zimpro公司开发的湿式空气氧化法(Wet air oxidation)已成功地用于城市污水污泥、造纸黑液、石油化工等高浓度有毒有害废水的处理, 它一般需高压和较高温度的条件, 投资费用高. 自60年代Eisenhauer研究使用Fenton试剂处理苯酚废水和烷基苯废水之后, Fenton试剂在工业废水处理中的应用研究受到国内外的普遍重视, 它特别适用于难生物降解或一般化学氧化难以奏效的有机废水处理, 该方法的最大问题是处理成本过高, 目前还难以推广[1,2]. 本研究在上述2种方法基础上探讨一种能有效处理高浓度难降解有机废水的新方法——低压湿式催化氧化法(Low pressure wet catalytic oxidation, 简称LPWCO), 该法主要是利用硫酸和Fenton试剂的协同作用, 使废水中有机物在较低的温度和压力下进行湿式催化氧化、炭化处理, 以推进湿式氧化法的工业进程.

## 1 实验方法及分析

### 1.1 主要设备仪器和原料成分分析

自制不锈钢低压反应釜(< 1.6MPa), 含酚废水用化学纯苯酚配制, 98%硫酸、硫酸亚铁和双氧水(30%)均为化学纯. 水样中苯酚采用4–氨基胺替比林比色法和LC–6A高效液相色谱仪(日本)分析, COD用重铬酸钾法分析, 铁用WYX 401原子吸收分光光度计测定.

### 1.2 实验方法

先用高浓度苯酚进行实验, 然后再用高浓度染料、农药水样进行实验. 实验中将一定浓度苯酚、硫酸、硫酸亚铁和过氧化氢加入反应釜中, 再用蒸馏水定容, 反应液为釜容积的1/2–1/3(保证液相反应), 密封反应釜, 加热升温升压, 反应后, 降温冷却取样分析.

## 2 含酚废水处理结果与讨论

### 2.1 过氧化氢浓度的影响

取一定量的含酚水样(酚6000mg/L, COD = 14350mg/L), 加入硫酸(控制$H_2SO_4$浓度为0.939mol/L, 即9.2%)、$FeSO_4 \cdot 7H_2O$(使水样中铁离子量达$1.21 \times 10^{-3}$ mol)、不同量的$H_2O_2$. 反应在0.6MPa(表压)165℃下恒温恒压反应1h, 反应后取样分析, 结果如图1所示.

由图1可见, 就去除苯酚而言, LPWCO法与Fenton法一般都在85%以上, 但对去除COD而言, 在$H_2O_2$·COD为0.2–1.4范围内, LPWCO法比Fenton法的效果要高20%–40%, 反应后水样由白色变为棕黄色, 水样中出现黑褐色沉淀, 此沉淀一部分沉入水底, 一部分浮于水面, 随着$H_2O_2$·COD的比值增大, 浮于水面的黑褐色

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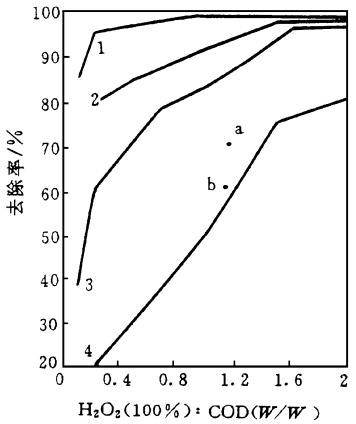


图1 $H_2O_2$投量与 COD、苯酚去除率关系

1. LPWCO 法酚去除率 2. Fenton 法酚去除率

3. LPWCO 法 COD 去除率 4. Fenton 法COD 去除率

悬浮物逐渐增多; 在 $H_2O_2$·COD 比值大于1.4后, Fenton 法对 COD 的去除率随 $H_2O_2$投量的增加而增大较多, 而 LPWCO 法则增加不多, 最后2条曲线趋于接近.

Devlin[3] 等人研究了水中苯酚的化学氧化机理, 可粗略地描述为: 苯酚–苯醌–马来酸–乙醛酸、乙二酸、丙二酸–丙酸、乙酸、甲酸–$CO_2$ + $H_2O$, 浓硫酸对纤维素、糖、脂肪碳水化合物有很强的脱水能力, 造纸黑液中的主要污染物是木质素, 木质素是以芳香结构为主体含有酚羟基的大分子化合物, 在加压加温(0.1– 0.6MPa, 104–165℃) 条件下稀硫酸(5%– 10%) 能使其脱氢(脱水)、聚合(缩合)、炭化[4]. 活性污泥的组分以脂肪结构为主, 也具有一定的芳香结构, 在同等的温度压力条件下, 稀硫酸(5%– 10%) 也能使其脱氢、聚合、炭化. 同样, 在此条件下稀硫酸也能使水中的苯酚、苯醌类有机物脱氢、聚合、炭化.

LPWCO 法处理含苯酚水时, 在 $H_2O_2$ ·· COD 值较低(0.2– 0.8) 阶段, Fenton 试剂与硫酸的共同作用, 使苯酚、苯醌等较大分子有机物脱氢聚合炭化, 成为大颗粒从水中沉淀出来, 使 COD 降低. 所以与 Fenton 相比, LPWCO 法对 COD 的去除率提高很多, 达40%左右. 在 $H_2O_2$ ·· COD 值较高时(0.8– 2.0), 由于前期 Fenton 试剂与硫酸共同作用, 水中绝大部分苯酚、苯醌类大分子有机物已除去, 剩下的有机物大部分是小分子的甲酸、乙酸等, 由它们构成的 COD 只能靠 Fenton 试剂中的羟基自由基的氧化分解作用才能除去. 所以, 在 $H_2O_2$: COD 比值较高阶段 LPWCO 法与 Fenton 试剂法的效果趋于接近, 仅相差20% 左右.

图1中 a 点为往含酚废水中投加 $H_2O_2$ + $Fe^{2+}$ + $H_2SO_4$时的 COD 去除率; b 点为只投加 $H_2O_2$ + $Fe^{2+}$ 时的 COD 去除率. 由此比较看出硫酸与 Fenton 试剂的协同效应.

**2.2** $Fe^{2+}$ 浓度的影响

试验条件基本同2.1. 固定水样 $H_2O_2$浓度为 0.491mol/L, $H_2O_2$ ·COD 为1.15, 只改变 $Fe^{2+}$ 的浓度, 结果见图2.

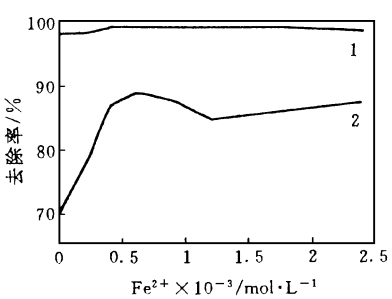


图2 $Fe^{2+}$ 浓度与 COD、苯酚去除率关系

1. 酚去除率 2. COD 去除率

由图2可见, 在 LPWCO 中 $Fe^{2+}$ 浓度对酚去除率影响不大, $H_2O_2$本身就是强氧化剂, 在加温加压和 $H_2SO_4$存在的条件下, 其氧化能力本身就很强, 很容易破坏苯酚的羟基, 即使不加 $Fe^{2+}$, 酚的去除率也高达98%, 而 $Fe^{2+}$ 浓度对 COD 去除率影响较大, 未加 $Fe^{2+}$ 时, 双氧水及硫酸的协同作用使 COD 去除率达70% 左右, 随着 $Fe^{2+}$ 浓度增高至 $0.6\times10^{-3}$时, COD 去除率增高20%, 以后 $Fe^{2+}$ 增高对 COD 去除率影响不大, 这表明在此条件下 $Fe^{2+}$ 起到了催化作用, 它使过氧化氢催化分解成羟基自由基(·OH), 羟基自由基具有比双氧水高得多的氧化电极电位.

$Fe^{2+} + H_2O \rightarrow Fe^{3} + HO\cdot + HO^-$

$HO\cdot + H^+ + e \rightarrow H_2O$ (标准电极电位 2.80V)

$H_2O_2 + 2H^+ + 2e \rightarrow H_2O$ (标准电极电位

1.77V)

所以, Fenton 试剂中的 $H_2O_2$ 比单独的 $H_2O_2$ 氧化能力更强, 对 COD 去除率更高. 水中 $Fe^{2+}$ 浓度增高有利于 $Fe^{2+}$ 与 $H_2O_2$ 的分子碰撞, 发生催化分解, 但 $Fe^{2+}$ 达到一定浓度后, 这种作用影响就不大了.

反应前后水样中铁离子用原子吸收分光光度计进行定量分析, 结果表明铁离子的总量未发生变化, 同时对水样中黑褐色的沉淀物进行定性分析, 也未发现含铁, 再次证明在 LPWCO 法中 $Fe^{2+}$ 起的是催化作用.

**2.3**　硫酸投量的影响

试验条件基本同上, 铁离子浓度固定为 $0.604\times10^{-3}$ mol/L, 投入不同量硫酸, 进行 LPWCO 试验, 结果见图3.

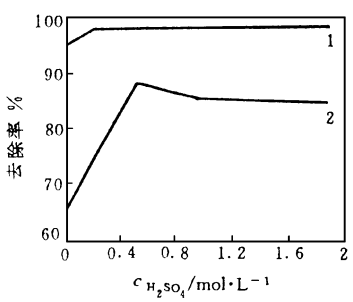


图3　$H_2SO_4$ 浓度与 COD、苯酚去除率关系

1. 酚去除率　2. COD 去除率

由图3可见, 未投加硫酸时, 同样的加温加压条件, Fenton 试剂对 COD 的去除率为64.4% (常温时为56%), 投加硫酸后, 由于硫酸的脱氢、聚合和炭化作用, 其 COD 去除率随硫酸投量的增加而增大, 但硫酸的脱氢、聚合和炭化作用对大分子的碳氢化合物(如芳烃)有效, 而对小分子的甲酸、乙酸则不起作用, 故硫酸投量增大到一定量后, 由于 $H_2O_2$·COD 值固定, COD 去除率不再增高.

对试验反应前后水样中的 $SO_4^{2-}$ 用钡盐法分析, $SO_4^{2-}$ 总量也未发生变化, 结果表明 $H_2SO_4$ 在此起的也是催化作用.

**2.4**　温度压力的影响

取一定量不同浓度含酚水样, 固定硫酸浓度为0.563mol/L, $Fe^{2+}$ 浓度为 $0.604\times10^{-3}$ mol/L, $H_2O_2$ 浓度为0.491mol/L, 在不同温度压力下恒温恒压反应1h, 冷却后取样分析, 结果如图4.

由图4可见, 反应压力(温度)对 COD 去除率影响不显著, 这说明在此条件范围内压力(温度)不是 LPWCO 法的决定因素, 决定因素仍然是 $H_2O_2$·COD 的比值. 用图4中压力为0.6MPa 所对应的1、2、3、4条曲线中 COD 去除率与图1中曲线3对应点比较, 两者基本吻合, 相差不大, 再次说明在一定的压力(温度)、硫酸浓度、铁离子浓度、反应时间条件下, 影响 COD 去除率的主要因素是 $H_2O_2$·COD 的比值大小.

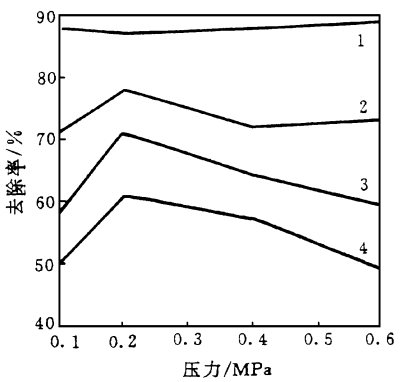


图4　压力(温度)与 COD 去除率的关系

1. COD= 14350mg/L $H_2O_2$·COD= 1.16 $H_2SO_4$·COD= 3.43

2. COD= 35870mg/L $H_2O_2$·COD= 0.481 $H_2SO_4$·COD= 1.53

3. COD= 71750mg/L $H_2O_2$·COD= 0.232 $H_2SO_4$·COD= 0.76

4. COD= 107600mg/L $H_2O_2$·COD= 0.155 $H_2SO_4$·COD= 0.51

## 3　染料、农药等废水试验

配制各种浓度的染料、农药等难降解的高浓度废水, 按2.4条件(固定反应压力0.6MPa, 164℃)进行试验, 结果见表1.

表1　**LPWCO** 法处理染料、农药等废水试验结果

| 水样 | 浓度 /g·L⁻¹ | 反应前COD /mg·L⁻¹ | 反应后COD /mg·L⁻¹ | COD 去除率 /% |
|---|---|---|---|---|
| 活性艳红 | 5.0 | 3450 | 720.6 | 79.1 |
| 亚甲基兰 | 5.0 | 5158 | 1269 | 75.4 |
| 十二烷基硫酸钠 | 10.0 | 21400 | 5154 | 76.0 |
| 甲胺磷 | 11.0 | 9418 | 1643 | 82.6 |
| 间苯三酚 | 7.51 | 16590 | 1112 | 93.3 |

试验结果表明, LPWCO 法处理高浓度难降解有机废水的效果很好, 适用的污染物种类

也较多.

## 4 结束语

LPWCO 的作用原理可初步看作是基于加温加压下硫酸与 Fenton 试剂的协同催化作用, 适于高浓度难降解有机废水处理, 特别适用于农药、染料、石油化工生产中含硫酸的高浓度有机废水处理.

LPWCO 法处理高浓度难降解有机废水的适宜条件为: $H_2O_2$·COD(重量比) = 0.2– 1.0, $Fe^{2+}$ = 0.6 × $10^{-3}$ mol/L 左右, $H_2SO_4$ = 0.5mol/L 左右, 废水 COD> 1×$10^4$mg/L, 操作压力0.1– 0.6MPa, 温度104– 165℃.

与 Fenton 试剂法相比, 本法在 $H_2O_2$·COD = 0.2– 0.8范围内 COD 去除率提高40%左右, 故将该法作高浓度难降解有机废水的预处理, 然后再进行生化处理, 将是一种有效实用的处理工艺. 该工艺避免了 Fenton 法 $H_2O_2$消耗过高的问题, 废水中剩下的 COD 主要是小分子易降解有机物(如甲酸、乙酸、丙酸等), 可用廉价的生化法进行处理.

与湿式氧化法(WAO)相比, 本法虽然消耗了一些 $H_2O_2$, 但操作压力仅为 WAO 法的1/10, 设备投资费大大降低, 操作条件较易达到, 易于实现工业化.

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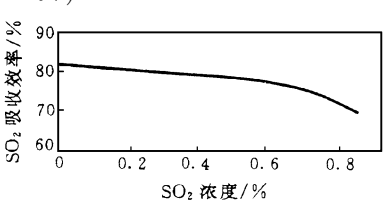


图8 制酸尾气中 $SO_2$浓度波动对$SO_2$吸收效率的影响
固液比: 1/10, 液气比: 0.6L/Nm³

表2 产品质量比较

| 项 目 | $MnSO_4$·$H_2O$/% | Fe% | pH |
|---|---|---|---|
| 国标二级[1)] | ≥95 | ≤0.008 | 5-7 |
| 本产品[2)] | 94.58 | 1.78 | 6 |

1) GB1622-86 标准 2) 按 GB1622-86检验

标, Fe 超标, 这主要是因为产品制取过程未设置氧化除铁所致. 若设置专门工序除铁, 用软锰矿($MnO_2$) 氧化母液中 $Fe^{2+}$ 为 $Fe^{3+}$ 后再中和及静置沉淀, 然后将母液过滤浓缩, 产品质量可望达到国家二级标准.

## 3 结论

(1) 软锰矿经还原再吸收$SO_2$生产$MnSO_4$·$H_2O$具有$SO_2$吸收效率高、Mn 利用率高及产品 $MnSO_4$·$H_2O$ 含量高等特点, 用于处理低浓度 $SO_2$是完全可行的.

(2) 所采用的软锰矿, 在最佳操作条件下, 还原效率可以保证大于94%. 用筛板塔吸收$SO_2$能保证反应充分、迅速. 产品制取不需复杂工艺. 因此, 该法具有工艺简单、操作控制方便、便于推广应用等优点.

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**Groundwater Pollution near Waste Ash Ponds of Coal-fired Power Plant —— A Case Study for $F^-$ Pollution in Paoche Ash Ponds of Xutang Power Plant, Jiangsu Province**. LiLi, Hai Huang (Dept. of Earth Sciences, Nanjing University, Nanjing 210093), Fahua Zhu (Nanjing Environmental Protection Research Institute for Electric Power, Nanjing 210031): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 59—61

A 2-dimensional pollutant transport model for groundwater pollution in the aquifer near ash ponds of Xutang Coal-fired Power Plant was established in this paper, and selecting $F^-$ as the simulating factor, the variations of groundwater hydraulic heads and $F^-$ concentration were simulated. At last, the polluted area and pollution degree of $F^-$ were predicted by the model. According to the simulating results, the model is reasonable, reliable and practicable. It provides a scientific method to predict the polluted area and pollution degree caused by the waste ash ponds.

**Key words**: groundwater pollution, pollutant transport model, numerical simulation, pollution prediction, coal-fired power plant.

**A Study of Pulsed Corona Discharges for Methlene Chloride Destruction**. Zheng Lei and Jiang Xuanzhen (Dept. of Chemistry, Zhejiang University, Hangzhou, 310027): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 62—64

in this paper, High voltage pulsed corona Discharges has been used for destruction of methlene chloride with concentration of 42.8μmol/L in air. Both positive and negative pulse generators were tested and found that the positive one can give much higher destruction efficiency than that on the nagetive one. The value of capacitors for pulse formation ($C_p$) and the material of electrodes also influence the destruction efficiency. A packed bed corona reactor with 2—3 mm spherical $BaTiO_3$ pellets as a catalyst was used in this experiments. Enhancement of $CH_2Cl_2$ destruction and the conversion of 90% were demonstrated. It may be attributed to the partial corona discharge induced by the contacted points between $BaTiO_3$ pellets, and then the density of corona was enchanced. The corona and catalyst combined technology gives a better destruction efficiency compared with that without $BaTiO_3$ catalyst.

**Key words**: pulse corona discharge, destruction, $CH_2Cl_2$, $BaTiO_3$.

**Comparison of Effect for Removing Mutagens and Inoganic Ions in Tap Watar by Revese Osmosis and Nanofiltration**. Li Lingzhi (Dept. of Chem., Pingdingshan Teachers College, Henan, 467002), Zhou Rong and Wang Zhansheng (Dept. of Environ. Eng., Tsinghua Uni., Beijing 100084): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 65—67

In order to get superior drinking water, tap water was treated respectively by reverse osmosis (RO) and nanofiltration (NF) in the laboratory. The removal effects of mutagens and ions by Ro and NF were compared. The Ames test results showed that both RO and NF could convert mutagenicity from positive to negative, while the ions removal effects of the RO and NF membranes are different, the removal rate of one-valence positive ions ($Na^+$, $K^+$) by NF is tenpercent lower than that by RO, the removal rate of two valence positive ions ($Ca^{2+}$, $Mg^{2+}$) is a little lower than that by RO. More ions which are beneficial to human health pass through nanofiltration mombrane into drinking water.

**Key words**: reverse osmosis, nanofiltration, advanced water, mutagens, inoganic ions, Ames test.

**Study on Wet Desulphurization with Pyrolusite to Produce $MnSO_4 \cdot H_2O$ in Smelting Plant**. Ning Ping, Sun Peishi et al. (Dept. of Environ. and Chem. Eng., Kunming University of Science and Technology, Kunming 650093): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 68—70

An additional experiment, in which reduced pyrolusite (made in laboratory) is used as absorption agent in wet desulphurization to produce $MnSO_4 \cdot H_2O$, has been done in a foam tower at a smelting plant. Optimum conditions for both reduction of pyrolusite and absorption of $SO_2$ are obtained and pure of 95% $MnSO_4 \cdot H_2O$ has been produced by primary crystallization of the absorption mother liquor.

**Key words**: sufur dioxide, reduced pyrolusite, wet desulphurization, smelting gas, foam tower.

**Study on the Low Pressure Wet Catalytic Oxidation Treatment of High Concentration and Refractory Organic Wastewater**. Yang Runchang, Zhou shutian (Dept. of Environ. Eng., Dept. of chem. Eng. Xiangtan University. Xiangtan 411105): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 71—74

Based on catalytic wet air oxidation and Fenton reagent, a new wet catalytic oxidation (LPWCO) method, which requires low pressure for the treatment of high concentration and refractory organic wastewater was studied. The method compared with general catalytic wet air oxidation, the pressure of the treatment is 0.1—0.6MPa, and the latter is 3.5—10MPa. In addition, its temperature is

no more than 180℃. The removal of COD by the treatment is over twenty percents more than Fenton's, while, $H_2O_2$ ··COD (weight ratio) less than one point two at the condition of phenol influent content more than 14000mg/L of COD. The existence of synergistic effect for COD removal in $H_2SO_4$+ Fenton system under the condition of added pressure and heating(0.1– 0.6MPa, 104– 165℃) was verified. It was carried out that five kinds of dye and pesticide wastewater was treated using the method.

**Key words**: wet oxidition, Fenton reagent, catalytic oxidation, organic wastewater, wastewater treatment.

**Study on the Detoxication Effect of Chromate Sludge by Red Brick Method with Shale Rock–Clunch**. Yang Guang et al. (Research Center of Resources Comprehesive Utilization Eng., Chongqing University 630044): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 75—77

The chromate sludge brick was made using shale rock, chromate sludge and clunch as main materials. When directions for producing materials are 20% of chromate sludge and 10% of clunch. The determination of whole brick powder showed that detoxication of Cr (VI) is thorough and stable; and determination of brick surface layer powter showed that the leaching concentration of water soluble Cr (VI) is 1.16mg/L. through five years following trail of tests under the free air and sun conditions Cr (VI) concentration can still achieve the standard of GB5086–85. The detoxication effect is mainly influenced by kiln temperature, acid–alkali property of the system, coal content and auxiliary.

**Key words**: chromate sludge, brick manufacture, red brick method, shale rock, clunch, detoxication.

**Spectrophotometric Method for the Simultaneous Determination of Phenols and Aromatic Amines in Sewage with Extraction–Reextraction**. Li Meirong, Yuan Cunguang et al. (Dept. of Chem. Eng., University of Petroleum, Shandong, 257062): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 78—80

This paper deals with a method of simultaneous determination of phenols and aromatic amines which extracted by ether, then reextracted by 10% NaOH and 10% HCl respectively. The sensitivity is improved highly, and many kinds of interferences is removed efficiently. Phenols of 0.03– 6.0 mg/L and aromatic amines of 0.008– 0.5mg/L can be determined.

**Key words**: phenols, aromatic amines, extraction–reextraction, double–wavelength, spectrophotometry, ether.

**The Application of Artificial Neural Network in Chinese Environmental Forecast**. Wang Ying and Sang Dayong (Dept. of Aeronautial Management Engineering, The Air Force Institute of Engineering, Xian 710038), Sun Linyan (School of Management , Xian Jiaotong University, Xian 710049): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 81—83

According to suitability of models for environmet forecast, a mutiple layer perceptron environment forecast model was built using artificial neural network as a new forecast method, with which the environment indices of 2000 were forecasted based on environmental data and economic data in 12 years (1981– 1992). Future strategies were also analyzed on the basis of forecasted data.

**Key words**: environment forecast, artificial neural network, mutiple layer perceptron environment forecast model.

**Data Acquisition for Inventory Analysis in LCA**. Xi Deli, Peng Xiaoyan (Dept. of Environ. Eng., Tsinghua University, Beijing 100084): *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 84—87

The LCA inventory analysis is an important stage in LCA after its scope and goal are defined. According to the real situation in China, a set of methods of data acquisition for life cycle inventory analysis were developed in this paper. It also gave concrete procedures for obtaining the social data of products through pollution coefficients of industrial departments and for gathering and checking data from enterprises by using production mass scheme respectively.

**Key words**: life cycle assessment, inventory analysis, data acquisition.

**Viewpoint on the Air Resources**. Ning Datong, Yuan Jun et al. (Institute of Environ. Sci., Beijing Normal University, Beijing 100875), *Chin. J. Environ. Sci.*, **18**(5), 1997, pp. 88—90

It is proved that air resource is one of the most valuable natural resources by means of analyzing and expounding. From the standpoint of atmospheric environmental carrying capacity for pollutants, the ambient air quality is divided into two parts of "quidditative" and "heterogeous", and its method of assessment is approached. On the basis of analyzing the air resource's value, a preliminary solution in measuring its value is also given in this paper. Furthermore, the effective ways for air resources protection are studied.

**Key words**: air resources, value, quality assessment, atmospheric environmental carrying capacity for pollutants.