膨润土吸附-絮凝法处理废水中的有机染料

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摘要 研究了天然膨润土对阳离子、分散、还原、中性、活性和直接等类有机染料的吸附特性,并比较了膨润土吸附-絮凝法与单纯絮凝法处理染料水溶液的脱色效果,前者比后者的脱色率可提高40%—200%。使用0.01%的膨润土加0.005%的聚合氯化铝,可使以阳离子染料为主的印染废水脱色率达94%—100%。

关键词 膨润土,吸附,絮凝,废水处理,有机染料。

膨润土是以蒙脱石为主要组分的粘土。蒙脱石是2:1型层状硅铝酸盐,其结构单位层是由2个Si—O 4面体晶片和它们之间夹着的一个Al—O或Al—OH 8面体晶片组成。由于晶格置换面产生了永久性的结构电荷,这种负电荷为交换性阳离子所平衡,不受价质pH值的影响,是蒙脱石电负性的主要来源。此外,由于离子吸附和晶格离解而产生的电荷属于端面电荷,其密度和符号随介质pH值的变化而改变,在总电荷中所占的比例一般很小[1]。除了上述基于静电因素的吸附外,尚有表面络合等专性吸附;能降低表面能的物质所发生的物理性正吸附以及化学吸附(化学沉淀)等[2]。可见,实际吸附过程相当复杂,而就蒙脱石对阳离子的吸附而言,一般以离子交换作用为主。

国外对膨润土吸附染料的研究已有报道[3—6],本文目的在于研究国产膨润土对水溶液中有机染料的吸附特性和条件,并给出膨润土吸附-絮凝法处理印染废水的初步试验结果。

1 实验

1.1 试剂与仪器

膨润土产自山东胶州市,经X-射线衍射分析表明为含钙型蒙脱石。按文献[5]的方法测得土样的阳离子交换容量(CEC)为63.06meq/100g土,pH值为7.5,膨润值为12.6。

染料主要由青岛染料厂提供,部分购自化工商店。聚丙烯酰胺(PAM)为南中化学塑料试验厂出品,分子量约3×10^6;聚合氯化铝(PAC)为化工商店商品,结晶氯化铝含量≥87.5%。

主要仪器有SHA-C型水浴恒温振荡器,TDL-4型和LD5-2A型离心机,pHS-2型精密酸度计,72G型光电分光光度计。

1.2 温度条件的预实验

用咸性紫5BN、分散深蓝RB和中性枣红GRL 3种染料的0.01%水溶液分别在10℃、15℃、25℃、35℃进行2h的膨润土(0.1%)吸附脱色试验,结果表明,脱色率在此温度范围内没有明显差异。

1.3 关于吸附速度的预实验

称取2.5g膨润土,加入已预先恒温至25±0.5℃的2500ml含染料0.01%的水溶液中,开动搅拌,每隔10min抽取10ml悬浊液,离心,过滤,测定滤液中剩余染料浓度。结果表明,25℃时各种染料在30min内均可达到吸附平衡,其中碱性染料吸附最快,中性染料吸附较慢,分散、还原、活性染料的吸附速度介于上述二者之间。

1.4 吸附率(%)-pH关系的测定

在一系列磨口锥形瓶中分别加入一定量膨润土和已用酸碱调至某一pH值、一定浓度的待测染料水溶液50ml,在25±0.5℃下振荡60min后,离心、过滤,用分光光度计在相应的最大吸收波长处测定滤液和未处理原液的吸光度。

1.5 吸附等温线的测定

在一系列磨口锥形瓶中分别加入0.1000±

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1993年4月20日收到修改稿

0.0005g 膨润土和 50ml pH 值一定而浓度不同的某一染料溶液，在 25±0.5℃恒温水槽中振荡 60min 后，离心，过滤，取滤液用分光光度法测染料浓度。

2 结果与讨论

2.1 溶液 pH 值对膨润土吸附染料的影响

关于吸附率-pH 关系测定的实验分 2 组进行。一组为低浓度吸附，如图 1 所示。溶液中膨润土加入量为 10mg/mg 染料；另一组为高浓度吸附，如图 2 所示，对溶液中每 mg 染料加入 0.3—0.8mg 膨润土。结果表明，高浓度吸附的脱色率明显低于低浓度吸附的脱色率，这是由于蒙脱石的吸附容量有限。

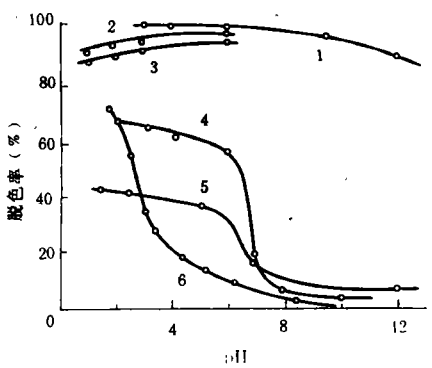

图 1 低浓度吸附时 pH 值的影响

1. 碱性紫 5BN 2. 分散黑 2BL 3. 分散大红 3GFL 4. 中性橙 RL 5 中性枣红 GRL 6. 大红色基 G

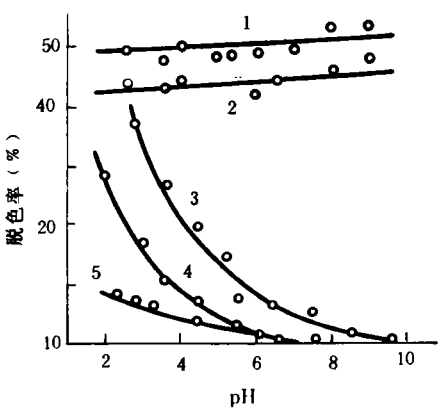

图 2 高浓度吸附时 pH 值的影响

1. 士林绿 2. 分散黑 2BL 3. 直接大红 4B 4. 中性橙 RL 5. 活性红 KD-G

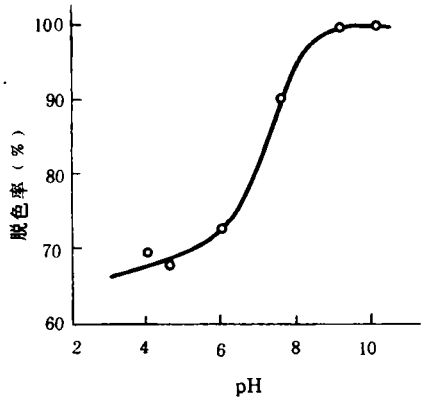

图 3 高浓度吸附时 pH 值对碱性紫溶液脱色率的影响

溶液 pH 值对膨润土吸附染料的影响可分为两类。一类如分散染料和还原染料，它们的水溶性极低，主要呈细颗粒分散于水中，在吸附过程中易附在土粒上而沉降，脱色率相对较高且与 pH 值关系不大。另一类是水溶性且分子结构中含有胺基染料，如中性橙、直接大红、活性红、大红色基等。这类染料 可能因为胺基是亲质子基因的关系，当 pH 减小时，容易被吸附到带负电荷的蒙脱石颗粒上，脱色率随 pH 减小而升高。由图 2 可见，在低浓度吸附时，中性橙 RL 需在 pH<6 时才能较明显地被吸附；而在高浓度吸附时；中性橙 RL、直接大红 4B 和活性红 KD-G 的水溶液都只有在 pH<3 时才有较明显的脱色。

碱性紫 5BN 的吸附情况很特别，它在低浓度吸附时，脱色率随 pH 值增大而略有降低（图 1）；在高浓度吸附时（1.6mg 膨润土/mg 染料），脱色率却随 pH 增大而明显升高（图 3）。这与该染料的分子结构 和蒙脱石的吸附机理有关，将另文讨论。

2.2 膨润土吸附染料的等温线

膨润土对水溶液中不同染料的吸附等温线示于 4—7 图中。前 3 个图中，包括直接大红 4B、中性橙 RL、活性红 KD-G 和碱性紫 5BN 的等温线都属一般类型，其中碱性紫完全符合 Langmuir 吸附规律，与文献[4]报道的一致。活性红在膨润土上的饱和吸附量小，且在实验条件下达到饱和吸附时溶液中的平衡浓度已达 20mg/L 以上（其它活性染料也有类似情况），故不适合用膨润土

作脱除剂。直接大红和中性橙在膨润土上

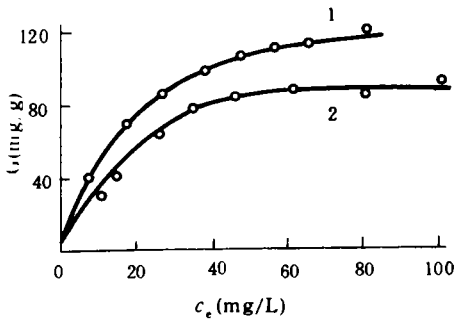

图 1　两种染料的吸附等温线

1. 直接大红 4B　　2. 中性橙 RL

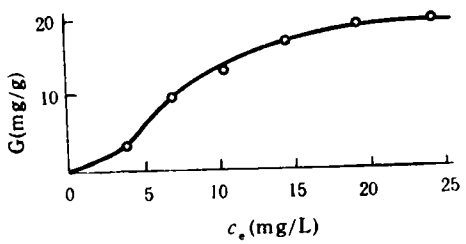

图 5　活性红 KD-G 的吸附等温线

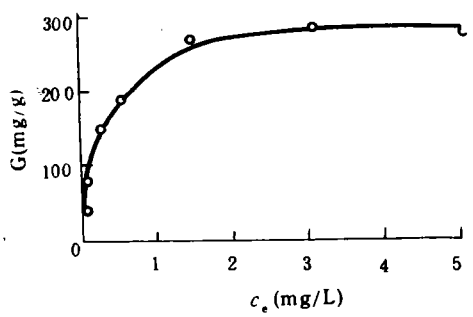

图 6　碱性紫 5BN 的吸附等温线

的饱和吸附量虽比活性红大，但在相同实验条件下达到饱和吸附时，溶液中的平衡浓度已达 60mg/L 左右，因此也不适宜用膨润土进行处理。只有碱性紫在膨润土上的吸附情况特别有利(图 6)，饱和吸附量高达 280mg/g 土，且在达到饱和吸附时溶液平衡浓度只有 2—3mg/L，因此，含有碱性紫或类似的阳离子染料污水，非常适合于用膨润土进行处理。

图 7 给出了分散黑 2BL 和士林绿的等温线，由于这 2 种染料的水溶性很低，它们主要是以微

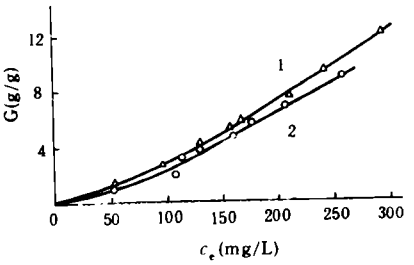

图 7　2 种染料的吸附等温线

1. 分散黑 2BL　　2. 士林绿

粒悬浮在水体中，加入膨润土，不过是产生了协同沉降而已，所以出现那种单调上升的曲线。用膨润土处理分散、还原 2 类染料有一定效果，但远不如处理碱性紫那样明显。

2.3　膨润土吸附-絮凝法与絮凝法效果比较

絮凝法是处理印染废水的方法之一[7,8]，对某些染料能够取得较好的效果。表 1 所示为膨润土吸附-絮凝法与单纯絮凝法的比较。其中染料浓度为 0.01%，每 mg 染料加膨润土 10mg；温度 20 ± 0.5℃；2 种方法的絮凝剂作用相同，PAC60mg/L，PAM10mg/L。

测定脱色率是待絮凝沉降后，吸取上清液测其吸光度与处理前原液的吸光度相比而得。吸附-絮凝法是在絮凝 1h 后测得，而絮凝法则是在絮凝 10h 后才得以测定(在本实验条件下，单纯絮凝法沉降速度相当慢)。

由表 1 数据可见，用膨润土吸附-絮凝法比

表 1　吸附-絮凝法与絮凝法的脱色率(%)比较

染　料	吸附-絮凝法	絮凝法
分散大红 3GFL	96.6	45.5
分散红玉 GFL	100	33.3
分散黄 5G	92.9	32.6
分散黑 2BL	98.1	28.3
分散草绿 S-2GL	96.6	66.7
分散深蓝 RB	96.2	60.0
碱性紫 5BN	99.3	40.5
	100[1)]	54.8[1)]
混合液[2)]	89.4	37.3

1)用 HPAM(水解聚丙烯酰胺)代替 PAM

2)以等量的碱性紫、2 种分散染料、2 种中性染料以及大红色基混合配制而成，最大吸收波长为 550nm

单纯絮凝法处理表1列出染料水溶液，脱色率可提高40%—200%。同时，絮凝速度加快。当然，对于单纯絮凝法，表1所用絮凝剂用量不一定是最佳的，通常要加入更多絮凝剂[7,8]，脱色率会提高一些，沉降速度也能加快，但改善幅度有限，不能改变上述基本结果。

膨润土吸附-絮凝法虽然对某些染料的脱色率不高，但其脱色效果不会随染料浓度减小而降低，甚至会有所提高；而单纯絮凝法对于很稀的染料溶液几乎没有效果，见表2。

表2 吸附-絮凝法(A)与絮凝法(B)对稀释染料溶液的脱色率比较[1)](%)

染 料	方 法	稀释倍数					
		3	4	5	6	10	20
大红色基A	A	17.6	16.2		17.6	18.4	20.4
	B	3.6	3.4		3.3	2.4	2.0
中性深黄GL	A	27.7	31.4		28.2	28.6	33.0
	B	8.4	6.7		5.6	2.4	0
中性橙RL	A	53.3	53.3	48.2	48.9	50.0	52.9
	B	13.0	12.0	10.7	10.6	8.8	5.9
中性枣红GRL	A	38.8	37.3	36.8	38.9	42.9	50.0
	B	15.7	13.3	11.8	5.6	2.9	0

1) 实试条件同表1

3 膨润土吸附-絮凝法处理印染废水初步试验

本试验的水样取自青岛第四针织厂，该厂生产废水主要来自腈纶、腈棉混纺、涤棉混纺以及少量棉纺针织品的染色废水，所用染料以阳离子染料为主，少量分散、活性等其它种类染料。

取水样1000ml置于大烧杯中，恒温至20±0.5℃，调pH至一定值，加入一定量膨润土，开动搅拌器搅拌一定时间后，加入60mgPAC，手持玻璃棒慢速搅动2min，静置1h后，在离液面1/3深度处吸取清液20ml，连同处理前的水样，一并用分光光度计测定吸光度。

第一次试验条件：pH＝6，9；吸附时间20min，30min；膨润土量400mg/L，100mg/L；絮凝剂PAC量100mg/L，50mg/L。用2个$L_4(2^3)$正交表交叉试验，以脱色率为衡量指标，结果十分接近：94.7%—97.9%。

第二次取样后将pH值调为6.0，再按上述条件用正交法进行试验，结果脱色率全部达到100%，于是将最佳条件定为：

pH＝6，吸附时间20min，膨润土用量100mg/L，PAC量为50mg/L。

对实采水样进行试验：

浅紫色，透明度较低，有少量悬浮物，pH≈6。最大吸收波长为540nm，在光程为10mm的比色皿中吸光度为0.158。COD_{Cr}＝187.05mg/L，BOD_5＝99.96mg/L。

经吸附-絮凝处理后，吸光度降为0，COD_{Cr}＝71.73mg/L，BOD_5＝27.05mg/L。即脱色率为100%，COD_{Cr}脱除率为61.7%，BOD_5脱除率为72.9%。

4 结论

(1)膨润土对有机染料的吸附机理主要在于蒙脱石的阳离子交换吸附特性，因此它最适合于处理主要含阳离子染料的印染废水，脱色率可达到或接近于100%。

(2)除还原、分散2类染料外，pH值对膨润土的脱除效果有很大影响。

(3)膨润土吸附-絮凝法处理印染废水的效果优于单纯絮凝法，在处理低浓度印染废水时，这种优越性愈加明显。

(4)膨润土吸附-絮凝法目前还有较大局限性，尤其不适用于高浓度的、成分复杂的印染废水，有待改进和完善。但该法成本低廉，操作简便，对某些印染废水有较好的处(下转第49页)

因此，要使床层内各点都处在扩散控制下操作，则加在反应器上的电压比理论计算值略大些。

2.3 工程因素对处理结果的影响

2.3.1 被处理液浓度

对一个按一定技术指标设计的反应器，有一最佳操作条件。当 $Cu10^{2+}$ 进口浓度发生变化时，必然会偏离优化点，当进口浓度高时，则使出口浓度偏高；当进口浓度偏低时，则使反应的 电流效率下降，能耗增加。为使反应器在最佳条件下操作，可使出口液部分循环使反应器的进出口条件满足优化设计的操作点 10[3]。

2.3.2 操作温度

理论上，温度升高反应器操作的槽电压可下降。实验发现，在其它条件不变时，反应器在10—40℃范围操作，槽电压的变化不明显，即温度对反应系统的影响不大。

2.3.3 操作电压

经优化设计的反应器在操作过程中，整个床层处于传质控制，维持该操作条件有一个最小操作电压。当电压高于此值时，对 $Cu10^{2+}$ 的出口浓度无影响，但会使放 H_2 的副反应增加，造成电流效率下降，能耗增加。

2.3.4 流体流速

流速的影响见表 3。流速增大时，反应的单程转化率下降，因此为了使模拟废液达到排放标准，有一最佳操作流速。

2.3.5 模拟废液电导率

实验发现，提高电导率，可改善反应器的操作特性。但在实际操作过程中，电导率是由模拟废液自身性质决定的。因此，为了改善反应器的操作特性，可混入酸洗废水或加入一些助剂来提高电导率，以提高处理效率。

2.3.6 填充材料

填充材料可采用导电颗粒、金属网或其它导电材料，填充材料的几何形状对处理结果有一定的影响。实验发现，采用网状填充材料可改善固体相电极的电接触性能。

3 经济估价

用本设计和操作的固定床电化学反应器处理模拟含铜废水时，理论上沉积出 1mol 铜所需电量为 53.6A·h，实验实际耗电为 65A·h，电流效率为 80%。所用操作电压为 2.5V 左右，从而回收 1mol 铜耗电 0.16kW·h，折合电费 0.048

元，其所回收的铜为电解铜，价值 1.0 元以上。处理后 $Cu10^{2+}$ 出口浓度可小于 1ppm，这样既处理了废水，又可回收得到有用的金属。该法同样适用于含其它重金属离子的废水处理。

4 结论

固定床电化学反应器对于含重金属离子的废水处理是一种有效经济的方法，该法不仅能使废水达到国家排放标准（$Cu10^{2+}$ 含量≤1ppm），同时能回收得到相应的金属。该处理过程设备投资小，操作费用低，具有良好的发展前景。

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（上接第 45 页）理效果，因此具有良好的发展前景。

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absorption spectrometry, plasma mass spectrometry, X-ray fluorescence, neutron activation, etc. For each of most elements, two or more different, reliable analytic methods were adopted. A computer was used to process the data obtained by statistical method. Any group- outliers were rejected by usingGrubbs, T, Dixon, Multi-level comparative S method, average geometric variation- Grubbs test. The best estimated values were obtained by taking the averages of preferred averages of all the data and analytic data as the four central values. The best estimated values thus calculated and complied with the certified values rule are taken as standard values. An evaluation of stability of the samples was made by X- ray fluorescence spectrometry during one year period. The results indicated that the storage stability of this standard reference material was satisfactory.

Key words: environmental soils standard reference material, stability, minimal amount of sampling, comparison of precision, certified values analysis, four central values.

The Optimisation of Grey non- linear River water Pollution Control System Using A Two Level Method. Zhang Xiangwei (Water Quality Research Center of China Beijing 100044): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 25—30

This paper focuses on the water quality planning problem of grey non- linear river water pollution control system using ideas of the grey system theory. Grey non- linear model and a two level method have been developed, which not only can describe the imperfection of water quality planning imformation but also can provide a new approach of dealing with the higher order, higher dimension and non-linear water quality planning model. The major studies involved are of three aspects: (1) Grey convex set, grey convex function and grey convex programme are definited; (2) Kuhn- Tucker condition for grey non- linear planning model has been given and (3) the optimisation of grey non-linear river water pollution control planning model using a two level method.

Key words: grey system theory, water pollution control system, two level method.

Sediment Oxygen Demand in the Yuancun Reach of the Pearl River in Guangzhou. Liu Fuqiang, Qi Sang (Institute of Aquatic Ecoscience, Jinan University, Guangzhou 510632): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 31—35

The physical properties, characteristics of oxygen demand, proportions of chemical oxidation and biological respiration in the total oxygen demand of sediments from the Yuancun Reach of the Pearl River in Guangzhou, were studied in laboratory during mean water and high water seasons. Results indicate that the sediment only consumed oxygen initially over the first six hours in mean water season, while in high water season, the sediment continuoeusly consumed oxygen at a comparatively low rate. It is doubt less that chemical oxygen demand plays a main role in total oxygen demand in both hydrological seasons. At the same temperature and water flow rate, the rate of sediment oxygen uptake in high water season is significantly higer than that in mean water season. In addition, the rates of sediment oxygen demand are related to the temperature and flow rate and their equations are: $SOD_M = 0.4945 \times 1.0058^{T-20}$, $SOD_H = 0.6155 \times 1.0234^{T-20}$, $SOD_M = 0.1623 \times 1.0912$, $SOD_H = 0.2393 \times 1.0857$.

Key words: Pearl River, sediment, biological oxygen demand, non-biological oxygen demand.

Study on the Treatment of Chrome-Leather Scraps as a Resource: Ⅷ Toxicological Test of Feed Collagen Protein. Jiang Tingda, Zhang Chunping (Research Center for Eco- Environmental Sciences, Chinese Academy of Sciences, Beijing 100085): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 36—38

The feed collage protein powder has a $LD_{50} > 10g/kg$, and can be assessed to be in an actually nontoxic grade. It has an accumulative coefficient $K > 5.28$, which is considered to be weakly accumulative. The Ames test result of mutagenesis is negative, either with or without adding S- 9 mixed liquid. The micronucleus test and spermatozoon malformation test also showed a negative reaction when dosage exceeded 5g/kg.

Key words: feed collage protein, toxicology.

Kitchen Wastewater Treatment by Iron- Carbon Flocculating Bed. He Weiguang, Guan Yaochu et al. (Chemistry Department of Zhongshan University): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 39—41

Kitchen wastewater can be treated by iron—carbon flocculating bed equipment. It was found that this method can be used to remove the animal and vegetable oils, COD, and BOD from kitchen wastewater with high removal rates, for example, of 96%, 72.5% and 90%, respectively.

Key words: iron- carbon flocculating bed, kitchen wastewater.

The Adsorption- Flocculation Method Using Bentonite for Treatment of Organic Dye-Containing Wastewater. Hang Hu, Hu Bolu et al. (Dept. of

Abstracts

chem., Qingdao Univ., Qingdao 266071): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 42—45

The adsorptions of cationic, disperse, reducible, neutral, active and direct dyes on bentonite are studied respectively and the decolorization effects of treating dye-containing aqueous solution by using bentonite adsorption-flocculation method and only flocculation method are compared. The decolorization rate of the former will be 40% — 200% higher than that of the latter. Using 0.01% of bentonite with 0.005% of PAC can decolorize the dye-containing wastewater by 94% — 100%, where in the dye consists mainly of cationic dye.

Key words: bentonite, adsorption, flocculation, treatment of wastewater, organic dye.

Treatment of Effluent Containing Cu Ions by Means of a Packed Bed Electrochemical Reactor. Xu Wenlin, Wang Yaqiong (Taiyuan University of Technology, Taiyuan 030024): *Chin. J. Environ. Sci.*, 15(1), 1994, pp. 46—49

A treatment process for effluent containing Cu ions is studied by means of a packed bed electrochemical reactor which is designed using one dimensional reactor model. Experimental results show that such a process particularly suitable for the treatment of the diluent effluent and the treated water can meet the requirement of the outlet concentration $< 1 \times 10^{-6}$; the factors affecting the effluent treament process, such as Cu ions input concentration, operation temperature, operation voltage, solution flow rate, effluent conductivity and the kind of the packed materal, are also discussed. This process is economically viable and provides an effective way of removing trace metal based on the assessment of the experimental process.

Key words: effluent containing Cu ions, packed bed electrode, electrochemical reactor, wastewater treatment.

Studies on the Treatment of Pharmaceutical Wastewater with Upflow Anaerobic sludge Blanket. Cheng Yu et al. (The Research Institute of Environmental Protection for Pharmaceutical Industry of Shenyang 110026): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 50—52

In this paper, described were some research results about the treatment of wastewater from Vitamin-C, SD and glucose prodution processes at a normal temperature with upflow anaerobic sludge blanket. The reactor has a volume of 100 litres, at a fermentation temperature of 17 — 24℃ and the influent COD_{Cr} of 3000—5000 mg/L. The organic load was 3 — 6kg COD/(m^3 • d), the hydraulic retention time (HRT) was 18—24h and the rate of gas generation reached to 0.3m^3/kg COD with 70% mechane in the gas. The COD_{Cr} removal was up to 90%. All the experimental results were satisfactory. This process is found appropriate for treating the lower concentration pharmaceutical wastewater under the anaerobic condition.

Key words: upflow anaerobic sludge, blanket granular sludge, *methanosarina*, total volatile acid.

Survey on Municipal Domestic wastes Composting Technology in Mainland China. Chen Shihe (School of Environmental Engineering, Tongji University, / Shanghai 200092): *Chin. J. Environ. Sci.*, **15** (1), 1994, pp. 53—56

This article deals with a general survey on the composting technology for municipal domestic wastes in mainland China, including: (1) the history of composting technology for municipal domestic wastes: initial stage, development and research stage, and application stage; (2) studies on composting technology: microorganism characteristic in composting process, oxygen delivery mechanism, composting process, factors affecting composting process, special-purpose machinery, trial engineering of composting technology, and existing problem; (3) the trend of composting technology for municipal domestic wastes in mainland China. This article gives a detailed and complete description on the evolution, current status and prospects of composting technology for municipal domestic wastes in mainland China.

Key words: municipal domestic wastes, composting technology.

Monitoring Methods for Genetically Engineered Microorganisms in the Environment. Tong Yongyi (Institute of Microbiology and Epidemiology, Academy of Military Medical Sciences, Beijing 100071): *Chin. J. Environ. Sci.*, **15**(1), 1994, pp. 57—60

This paper deals with the monitoring principle and methods for genetically engineered microorganisms (GEM) in the environment, including the requirements of marker system on GEM for monitoring purpose, some recently developed marker systems and the monitoring methods for environmental GEM culture method, immunological method, genetic method, bioluminescent method and application of flow cytometry.

Key words: genetically engineered microorganism (GEM), environment, monitor.

Analysis of the Reversible Mechanism of Inhibition by Synthetic Organics in Anaerobic Digestion. Bian Rulin (Deparment of Environmental Engineering, Xi' an Institute of Metallurgy and Construction Engineering, Xi' an 710055): *Chin. J. Environ.*