净化去除酸性废水中不同价态砷的研究

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摘要　根据净化含砷废水的铁盐中和法,通过理论分析和实验表明,在 Fe/As=0.5—6.0 范围内,铁盐中和法对废水中 3 价砷的去除率较其对 5 价砷的去除率平均低约 60%,证实了废水中的 3 价砷较 5 价砷难于去除。氧化剂选择研究表明,对于含砷量高的酸性废水,采用漂白粉[Ca(ClO)₂]氧化其中的 3 价砷最为适宜。对于含砷 782.5mg/L,pH=1 的酸性废水,采用氧化-铁盐中和法经一级处理后,废水中含砷量即可低于 8mg/L,除砷率大于 99%,二级处理后出水中砷含量即可低于国家排放标准(0.5mg/L)。

关键词　酸性废水,净化处理,砷。

铁盐中和法是工业上常用的含砷废水的净化处理方法,即向废水中按比例投加铁盐,而后用石灰中和的除砷方法。铁盐中和法包括亚铁盐中和法和高铁盐中和法。实践表明[1,2],对于含有 3 价砷和 5 价砷的酸性废水,采用铁盐中和法净化处理,通常对 3 价砷的去除难以达到令人满意的效果。为此,本文依据砷化物的电化学特性及有关理论分析,对铁盐中和法净化处理酸性废水中不同价态砷进行了实验研究。旨在为改进现有工业废水处理工艺及设施、提高除砷效率,提供科学依据。
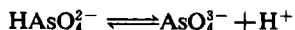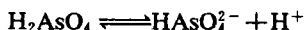
1　去除废水中 3 价砷和 5 价砷可行性分析

工业废水中由于同时含有氧化及还原性物质,因而酸性废水中溶解态砷的含氧酸的离解及其中砷的价态变化,对从废水中去除不同价态砷起着十分重要的作用。酸性废水中的砷主要以 3 价和 5 价砷的化合物形态存在。为了简化和便于理论分析,分别考虑废水中只存在 3 价砷或者 5 价砷的情况。

当废水中只存在 3 价砷(HAsO₂)时,其电离平衡式为:

$$HAsO_2 \rightleftharpoons AsO_2^- + H^+$$

当废水中只存在 5 价砷(H₃AsO₄)时,其电离平衡式为:

$$H_3AsO_4 \rightleftharpoons H_2AsO_4^- + H^+$$

$$H_2AsO_4^- \rightleftharpoons HAsO_4^{2-} + H^+$$

$$HAsO_4^{2-} \rightleftharpoons AsO_4^{3-} + H^+$$

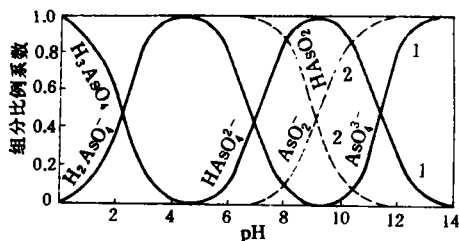根据以上方程式,通过电离平衡理论的分析和计算,可得出在不同 pH 值条件下,废水中 As(5+)化合物和 As(3+)化合物组分的变化情况如图 1 所示。

图 1　As 的各种离子随 pH 值的组分变化
1. As(5+)化合物　　2. As(3+)化合物

从图 1 可以看出,As(5+)在 pH 0—14 范围内均以砷酸及其离子的形态存在;而 As(3+)则在 pH=7 时才开始出现 AsO₂⁻ 的阴离子,而且 As(3+)由 HAsO₂ 分子转化为 AsO₂⁻ 的速度慢于 As(5+)各离子间相互转化的速度,即单位 pH 的变化,导致 AsO₂⁻ 阴离子的组分比例增大不多。这意味着 As(3+)要在较高的 pH 值下才能除去,而且较 As(5+)难于除去。

另一方面,根据胶体化学的原理,带电荷越多的离子越容易被胶体颗粒吸附去除。As(5+)的砷酸根离子 H₂AsO₄⁻、HAsO₄²⁻ 和 AsO₄³⁻ 分别带有 1—3 个负电荷,而 As(3+)的 AsO₂⁻ 只带 1 个负电荷,且在较高的 pH 值下才出现。这也表明 As(3+)较 As(5+)难于去除。

此外,根据溶度积沉淀理论,As(5+)能与一些金属阳离子生成溶度积很小的砷酸盐而易于被去除。如 $Ca_3(AsO_4)_2$:$K_{sp}=6.8\times10^{-19}$,$FeAsO_4$:$K_{sp}=5.8\times10^{-21}$,$Cu_3(AsO_4)_2$:$K_{sp}=7.6\times10^{-36}$,$Pb_3(AsO_4)_2$:$K_{sp}=4.1\times10^{-36}$,$Cd_3(AsO_4)_2$:$K_{sp}=2.2\times10^{-33}$。然而,As(3+)则不具备这种化学性质,这也决定了 As(3+)较 As(5+)难于去除。

根据以上分析,在废水除砷过程中,对3价砷施以适当的氧化剂,将其氧化成为5价砷而后除去,这是十分必要且合理的。

2 废水中不同价态砷去除的对比实验

2.1 实验方法

实验中 As(5+)和 As(3+)分别由 Na_3AsO_4 及 H_3AsO_3 水溶液配制。实验时取 1L 水样先用硫酸调节 pH 至 0—1,Fe^{2+} 和 Fe^{3+} 分别由 $FeSO_4$ 及 $FeCl_3$ 提供。加入铁盐并搅拌后,用石灰乳调节 pH 值到 7,再加入聚合硫酸铁絮凝沉降。

水样中的总砷采用二乙氨基二硫代甲酸银光度法测定,砷的价态分析采用正丁醇-醋酸乙酯一次萃取砷钼篮比色法测定[4]。总砷测定和砷的价态分析均在 721 型分光光度计上进行。水样的 pH 值由 pHS-2 型酸度计测定。

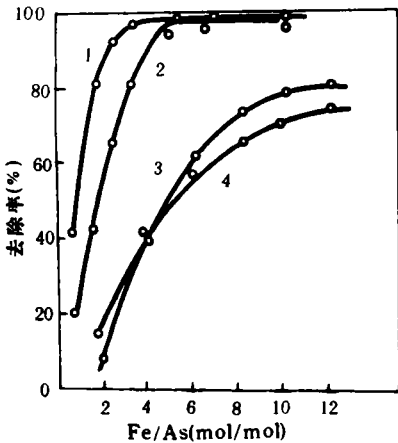### 2.2 Fe^{2+}、Fe^{3+} 铁盐对不同价砷去除效果对比

对比实验结果如图 2 所示。

图 2　Fe^{2+} 及 Fe^{3+} 铁盐对不同价态砷的去除效果

1. Fe^{3+},As^{5+}　2. Fe^{2+},As^{5+}　3. Fe^{3+},As^{3+}　4. Fe^{2+},As^{3+}

图 2 表明,铁盐中和法对酸性废水中的 As(5+)有较好的去除效果。当 Fe/As=1.3—2.2 时,As(5+)的去除率可达 90% 以上。此外,图中曲线还表明,Fe^{3+} 铁盐的除砷效果优于 Fe^{2+} 铁盐,这一差别在 Fe/As 低于 2 时较为明显。
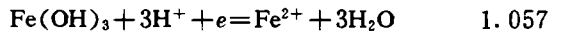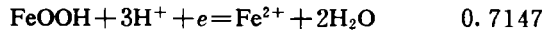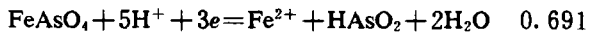
由图 2 可以看出,铁盐中和法对 As(3+)的去除效果远不如对 As(5+)的去除效果好。尽管在很高的铁砷比下,不论 Fe^{2+} 铁盐还是 Fe^{3+} 铁盐对废水中 As(3+)的去除率均低于 82%,且 2 种铁盐的去除 As(3+)的效果也相差不大。

2.3 废水中不同价态砷去除效果的对比分析

图 2 还表明,在 Fe/As=0.5—6.0 范围内,铁盐中和法对废水中 3 价砷的去除率较其对 5 价砷的去除率平均低约 60%,而且,无论是 Fe^{2+} 铁盐还是 Fe^{3+} 铁盐,对废水中 3 价砷的去除率均大大低于对 5 价砷的去除率。这也证明了前述 3 价砷较 5 价砷难于去除的理论推导。

3 氧化剂的选择研究

根据电化学理论,低价离子氧化成高价氢氧化物或氧化物的难易程度取决于其标准电极电位值 φ^0 的高低。电位越低越易于氧化水解。对于铁盐中和法除砷,其中一个重要的原因是铁离子属于低价易于水解而高价难于水解的一类离子,其半电池反应和氧化水解的 φ^0 值如下:[4]

$$FeAsO_4+5H^++3e=Fe^{2+}+HAsO_2+2H_2O \quad 0.691$$

$$FeOOH+3H^++e=Fe^{2+}+2H_2O \quad 0.7147$$

$$Fe(OH)_3+3H^++e=Fe^{2+}+3H_2O \quad 1.057$$

以上所列的 φ^0 值均小于或接近于 1(V)。由此可见,3 价砷和 Fe^{2+} 较易于氧化水解。因此,控制溶液中的电位就可选择性地进行氧化水解除砷,而溶液中的电位则取决于氧化剂的种类和溶液的 pH 值,浓度等条件。

目前工业上常用的氧化剂有 $S_2O_8^{2-}$、H_2O_2、O_3、MnO_4^-、ClO^-、Cl_2、HNO_3、MnO_2 等。从铁盐中和法除砷的技术和经济角度出发,本文选择漂白粉和软锰矿 2 种氧化剂,对酸性废水中 As(3+)氧化成 As(5+)进行实验研究。

3.1 氧化剂氧化能力的分析研究

在以漂白粉和软锰矿作为废水中 As(3+)

的氧化剂时,前者的有效成分为 $Ca(ClO)_2$,后者为 MnO_2。根据电化学原理并结合实际工业废水中存在 SO_2、H_3AsO_3 以及处理工艺中加入 Fe^{2+} 的情况,运用能斯特(Nernst)方程计算可求得相应的物质的电极电位与 pH 值的相互关系式:$\varphi = \varphi^0 + F(pH)$。如果将 $E = \varphi_{氧化剂} - \varphi_{还原剂}$ 定义为氧化还原反应中的推动力,则可以更加清楚地对 2 种氧化剂的氧化能力进行对比。图 3 中列出了 2 种氧化剂对含砷废水中相应物质氧化推动力与 pH 值间的关系曲线。

图 3　氧化剂的氧化推动力与 pH 值的关系
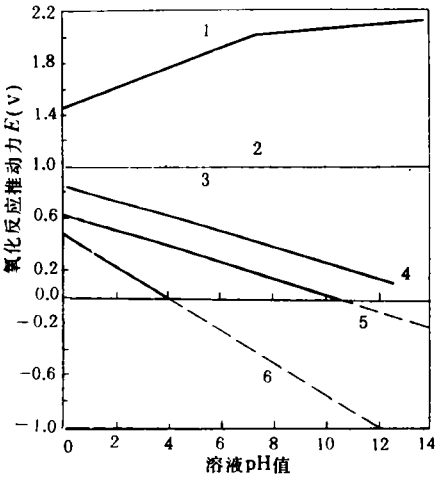1. $E(Ca(ClO)_2\text{-}SO_2)$ 2. $E(Ca(ClO)_2\text{-}H_3AsO_3)$ 3. $E(MnO_2\text{-}SO_2)$
4. $E(Ca(ClO)_2\text{-}Fe^{2+})$ 5. $E(MnO_2\text{-}H_3AsO_3)$ 6. $E(MnO_2\text{-}Fe^{2+})$

由图 3 可见,当加入 $Ca(ClO)_2$ 或 MnO_2 后,首先被氧化的是废水中的 SO_2,其次才是 H_3AsO_3,最后是 Fe^{2+},而且 $Ca(ClO)_2$ 对 H_3AsO_3 的氧化能力明显强于 MnO_2。需要注意的是,当 pH 值大于 3.9 时,$E(MnO_2\text{-}Fe^{2+})$ 为负值,说明 Fe^{3+} 可能会被还原为 Fe^{2+},这对除砷会产生不利的影响。当 pH 值大于 10.4 时,$E(MnO_2\text{-}H_3AsO_3)$ 也会出现类似情况。

3.2 氧化剂氧化能力的对比

为了验证氧化剂的氧化能力,采用某铜冶炼厂硫酸车间的含砷废水进行氧化实验。废水中的 3 价砷含量 2120mg/L,占总砷量的 82.8%。废水的 pH 值为 1 左右。根据有关的分析研究,对于废水除砷,pH=3.1—4.6 是一个较优的范围。因而选择 pH=1(直接氧化)和 pH=4(调节 pH 值后氧化)2 种 pH 实验条件。pH 值的调节采用 20% 的 NaOH 溶液,并用 $Ca(ClO)_2$ 和 MnO_2 进行氧化能力对比实验,结果如图 4 所示。

从图 4 看出,$Ca(ClO)_2$ 和 MnO_2 对废水中的 3 价砷均有氧化作用。然而,无论在 pH=1 或 pH=4 时,$Ca(ClO)_2$ 对 3 价砷的氧化效果均优于 MnO_2。当加入量大于 4.5g/L 后,$Ca(ClO)_2$ 对 As(3+) 的氧化率较 MnO_2 平均高出 4%—5%。尽管 2 种氧化剂在 pH=4 时对 3 价砷的氧化效果较 pH=1 时好,但从简化操作、降低费用角度出发,实际生产中以采用直接氧化(pH=1)为好。
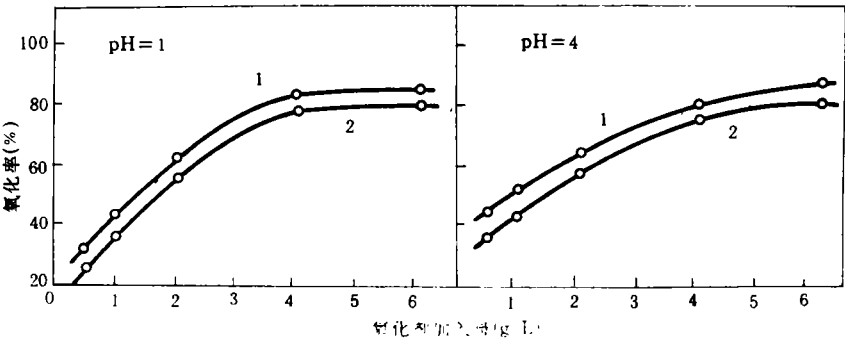

图 4　废水中 3 价砷的氧化实验结果
1. $Ca(ClO)_2$　2. MnO_2

4　现场实验结果

对某铜冶炼厂硫酸车间的高

因此,负荷率是综合反映进水浓度和水力停留时间影响的因素,它决定着供给反应器内微生物的食料量。显然,一项总因素的作用必然会大于2项分因素的作用,因而无论对COD去除率,还是对产气率,它都是最重要的。

上述分析得出的进水COD浓度的影响大于HRT的结论,从反应器中有机物的分解过程来看也是不难理解的。从理论上讲,在反应器中应当存在一个最小HRT值。在生物膜法中,有机物的分解过程是有机物首先从主体液体传递入生物膜,然后在生物膜内被微生物所降解。显然这里存在2个时段:一是有机物向生物膜内传递所需的时间,它主要取决于传质速率;一是生物膜内微生物降解传入的有机物所需的时间,它主要取决于降解速率(反应速率)。只要废水在反应器中的实际HRT大于这2个时段中的较长者,HRT的影响就不会很重要。微生物降解有机物属生物酶催化反应,酶催化反应速度比非催化反应速度高 10^8—10^{20} 倍,比其它催化反应速度高 10^7—10^{14} 倍[3]。所以只要微生物生长正常,降解速度是相当快的,也就是只要有机物传入生物膜中便可及时得到降解。而传质速度主要取决于主体液体的有机浓度、生物膜厚度以及反应器中的混合搅拌程度。在生物流化床中,只要膜厚小于

0.15mm,膜厚对有机物传递的阻力可以忽略[4]。本文所引试验数据的生物膜厚为 $92\mu m$,因此传质速率主要取决于主体液体的有机物浓度,而在一般二级处理所要求的废水排放浓度条件下(如COD100mg/L),传质速率可以保证较快。综上所述,废水在反应器中的HRT对有机物去除率的影响比进水COD浓度,就显得不那么重要了。

4 结论

(1)用灰色系统关联度方法分析厌氧反应器处理有机废水的效率影响因素,比其它分析方法简单,试验数据可以较少但其结果可靠。

(2)本文以厌氧流化床为例进行了计算得出:厌氧流化床在最适温度范围内处理有机废水时,影响其效率(COD去除率和产气率)的因素的重要性顺序为:负荷率、进水COD浓度、HRT。因此,设计和运行中首要应控制负荷率。

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砷烟气洗涤水进行了实验。首先向废水中按其含砷量的2.0倍加入漂白粉,将其中的3价砷氧化成5价砷,而后用石灰乳调节pH值至10左右,再按Fe/As=2.5加入硫酸亚铁除砷。

实验结果表明,对于含砷 782.5mg/L、pH=1 的酸性废水,采用氧化-铁盐中和法处理,经一级处理就可使废水中的砷含量降至 8mg/L 以下,二级处理后废水即可达标排放。

5 结论

理论分析和实验研究结果表明,在净化处理含砷废水的过程中,添加适宜氧化剂——漂白粉将其中的3价砷氧化成5价砷十分必要。这样做可以使现有含砷废水处理设施的除砷效果有较大幅度的提高。

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River. Yu Changrong et al. (Jilin Provincial Institute of Environmental Protection, Changchun 130012): *Chin. J. Environ. Sci.*, 15(4), 1994, pp. 35—38

Since the main industrial sources of mercury discharged into the Songhuajiang River were eliminated eight years ago, the total mercury and methylmercury contaminations in fish from the reaches of the river from Jilin City to Sanchahekou and from Sanchahekou to Tongjiang have been reduced to a light level of contamination and a medium level of contamination, respectively. The average levels of total mercury and methylmercury contaminations in fish from both reaches of the river exhibited: the highest in carnivorous fish, lower in omnivorous fish, and the lowest in herbivorous fish; higher in the underlayer fish than in the medium and upper layer fish; and higher in the scaleless fish than in the scaled fish. In the muscle of each species of fish from both reaches of the river, the level of methylmercury was in a significantly positive correlation with the level of total mercury while being in a significantly negative correlation with the activities of urease and lactate dehydrogenase (LDH). The level of total mercury in the muscle of fish was in a significantly multiple linear, multiple logarithmic or multiple exponential correlation with the level of total mercury in river water. The level of methylmercury in the muscle of fish was in a significantly multiple logarithmic correlation with the level of methylmercury in river water.

Key words: the Songhuajiang River, total mercury, methylmercury, contamination, fish.

Study on the Level of Mercury in Fishmen's Hair Since the Elimination of Major Sources of Mercury Pollution in the Songhuajiang River. Wu Shi'an et al. (Jilin Provincial Station of Sanitation and Antiepidemics, Changchun 130021): *Chin. J. Environ. Sci.*, 15(4), 1994, pp. 39—40

Since the major sources of mercury pollution in the Songhuajiang River were eliminated 8 years ago, the level of mercury in the hairs of fishmen living along the river has reduced by 78. 30%— 82. 5% as compared with that in the 1970s, and by 71. 48% as compared with that in the early 1980s, showing that obvious health benefit and environmental benefit have been obtained. However, it was found that so far the fishmen living along the river still have a significantly higher level of hair mercury than those living in a non-mercury polluted area, and 1. 92% of them have a level of hair mercury exceeding the national diagnostic standards. They were found to be mainly distributed along the first Songhuajiang reaches of the river downstream Sanchahekou. Extremely less number of cases for the fishmen living along the second Songhuajiang reaches of the river were found to have a level of hair mercury exceeding the national diagnostic standards, and the causes for this were discussed also.

Key words: mercury poisoning, water pollution, biological monitoring, Songhuajiang River.

Determination of Phthalic Esters in Aqueous Extracts from Solid Wastes. Dai Tianyou, Wang Shufang (China National Environmental Monitoring Center, Beijing 100012): *Chin. J. Environ. Sci.*, 15(4), 1994, pp. 41—43

The HPLC determination of phthalic esters in aqueous extracts from solid wastes were reported. In this study, a HPLC system equipped with a Zorbax-CN column and a 224 nm UV detector and using a n-hexane mixed with 0. 1% isopropanol as eluting agent was established and used to determine the phthalic esters in aqueous extracts from municipal refuses in Beijing. The system was effective to separate phthalic esters from non-polar organics in the extracts and to eliminate the interference likely resulted from some organic compounds which had a polarity similar to phthalic esters. Having been extracted with n-hexane and then concentrated, the extracts in organic phase could be directly analysed with the HPLC system without a further purification (e. g., column chromatography). Water samples fortified with phthalic esters at a level in the range of 1—500 ppb were analysed, with a linear response in the range of 0. 1— 450 ppb, a recovery for phthalic esters of 70%—110%, and the detectable limits of 1. 0 ng for dimethyl phthalate (DMP), 0. 4 ng for diethyl phthalate (DEP), and 0. 2 ng for each of di-n-butyl phthalate(DBP), di-n-octyl phthalate (DOP) and di(2-ethylhexyl) phthalate (DEHP), at 224 nm. The phthalic esters in aqueous extracts from several solid waste samples were found by using this system to be at a level of 5 ppb. The variation coefficient for 7 parallel tests on the extracts from solid wastes was within 10%.

Key words: phthalic esters, extracts, HPLC.

Study on the removal of Arsenic in Different States of Valence from Acidic Wastewater. He Shaoxian et al. (Dept. of Environmental Engineering and Chemical Engineering, Kunming Institute of Technology, Kunming 650093): *Chin. J. Environ. Sci.*, 15(4), 1994, pp. 44—46

Based on the process of iron salts neutralization to remove arsenic from wastewater, theoretical analysis and experiments indicated that, at a ratio of Fe/As = 0. 5—0. 6, As(III) was removed at a rate of about 60% lower than that of As(V), demonstrating that As(III) was more difficult to be removed from wastewater than As(V). In a further study, it was found that it was most suitable to oxidize As(III) in acidic wastewater containing a high level of arsenic by using a bleaching powder, Ca$(ClO)_2$. For an acidic wastewater containing 782. 5 mg/L of As at pH = 1, the oxidation/iron salts neutralization process used in a primary treatment produced an effluent containing less than 8 mg/L of As, with an arsenic removal of more than 99%. If followed by a further secondary treatment, the level of arsenic in the effluent produced would be lower

than 0. 5 mg/L, i. e. , the national standard for its discharge.
Key words: acidic wastewater, pollution control, arsenic (As), iron salts neutralization.

Study on the Manufacture of Activated Carbon from the Carbon in Coal Ash from a Power Station. Wu Xinhua and Yu Wei (Fujian College of Forestry, Nanping 353001): *Chin. J. Environ. Sci.* , **15** (4), 1994, pp. 47—49
A production process of activated carbon in which the carbon in a coal ash from a power station was used as a starting material has been developed. The optimized conditions for this process to produce a granular activated carbon were using charcoal, white charcoal or coal as an auxiliary raw material, together with which the starting material was undergoing a treatment in a preactivation process, then was washed with acid and water, and finally was activated or even further reactivated if required. The activated carbon product thus produced had an iodine value of 600— 700 mg/ g. The activated carbon from a pilot industrial production had an iodine value of 630— 800 mg/ g with a wearability of over 95%. This process provides a new way for coal ash to be utilized comprehensively.
Key words: coal ash, activated carbon, comprehensive utilization.

Comparative Study on the Capacities of Aerobic and Anaerobic Immobilized Microbes to Treat Organics. Wu Xiaolei et al. (Dept. of Environ. Eng. , Tsinghua University, Beijing 100084): *Chin. J. Environ. Sci.* , **15**(4), 1994, pp. 50 —52
Activated and anaerobic sludges were respectively immobilized with polyvinyl alcohol (PVA) used as an entrapping agent, and then the immobilized sludges were separately used to degrade the organics in wastewater under the aerobic and anaerobic conditions, respectively. Comparisons in the capacity of treating organics were also made between the immobilized and free sludges and between the immobilized activated sludge and the immobilized anaerobic sludge. The results show that the volumetric loading was 1. 3 to 2. 1 times that of free sludge, meant by that the immobilized sludges had a higher capacity of treating organics than a free sludge. Under the conditions studied, the volumetric loading ratio of the immobilized anaerobic sludge to the free anaerobic sludge (2. 13) was much higher than that of the immobilized activated sludge to the free activated sludge (1. 30— 1. 54). Considering the sludge loading and gas yield per unit of sludge by weight, it was concluded that the capacity of microbe treating organics could be fullier developed in the immobilized anaerobic sludge so that the immobilized microbes entrapped in a gel would be more suitable for the anaerobic treatment of a high strength organic wastewater.
Key words: immobilized microbes, immobilized activated sludge, immobilized anaerobic sludge, treating capacity.

Study on the Indicators for Evaluating the Activity of Immobilized Microorganism in the Degradation of Isocarbophos. Zhang Xiaohe et al. (Institute of Environmental Medicine, Tongji Medical University, Wuhan 430030): *Chin. J. Environ. Sci.* , **15** (4), 1994, pp. 53—55
The indicators for evaluating the performance of immobilized microorganism before and after the biodegradation of isocarbophos in water samples have been studied. It has been found that the levels and degradation rates of the organophosphorus pesticide in water were in highly positive correlation to COD_{Cr} and COD_{Cr} removal, respectively, so that it would be proper to choose COD_{Cr} removal as a routine indicator for evaluating the activity of immobilized microorganism in the degradation of this pesticide. What was given in this article also included the regression equations established on the basis of experimental data, and the results from their significance tests, wherein the correlative coefficients of Eqs. 1—4, Eqs. 5 and 7, Eqs. 6 and 8, and Eqs. 9—12 were 0. 992, 0. 940, 0. 951 and 0. 978, respectively.
Key words: Isocarbophos, immobilized microorganism, biodegradation, indicators for evaluating activity.

Speciation of Selenium in Soils. Lan Yeqing et al. (Dept. of Basic Courses, Nanjing University of Agriculture, Nanjing 210014): *Chin. J. Environ. Sci.* , **15**(4), 1994, pp. 56—58
The distribution of natural and applied selenium (Se) species in three kinds of soil, i. e. , tide-saline soil (C), gray tide-soil (G) and yellow brown soil (Y), in Jiangsu province was studied. The results show that the naturally occurred Se species were mainly distributed as residual species (F_5), and F_5 in each of the three kinds of soil accounted for about 80% of total Se species. After an incubation for 4 months, the applied Se species were relatively homogeneous to be distributed as soluble species (F_1) (except in Y), exchangeable species (F_2), aqueous ammonia extractable species (F_3) and residual species (F_5). With two different treatments, the distribution of Se species was found to be in some relation to soil pH value, glutinous grains and free iron oxide levels. The soil pH value was in such an order as $C \doteqdot G > Y$; the levels of glutinous grain and free iron oxides: $Y > C > G$; the percentage levels of F_1 and F_2 : $C \doteqdot G > Y$; and the percentage levels of F_3 and F_5 : $Y > C > G$.
Key words: selenium (Se), soil, species.

Preliminary Analysis of Design Flow for Allowable Discharge Capacity of Rivers. Zheng Yingming (Institute of Environ. and Water Conservancy, Hehai University, Nanjing 210024): *Chin. J. Environ. Sci.* , **15**(4), 1994, pp. 59—61

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