

让湖泊休养生息
全球挑战与中国创新

第十三届世界湖泊大会

论文集

中卷

中国环境科学学会 主编



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·北京·

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Preparation and Characterization of Representative Inorganic – PILCs Sorbent

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Abstract: Inorganic – pillared montmorillonites were used to adsorb heavy metal pollutant in Waste Water Treatment. In this paper montmorillonite pillared by mixing Fe, Fe – Cr, Al – Fe and Cu – Al complexes was preparation and was characterized by XRD, N₂ – BET, SEM and chemical analysis techniques. The XRD shows the d₀₀₁ of Fe, Fe – Cr, Fe – Al and Cu – Al montmorillonite were 15.6Å 15.8Å 19.8Å and 19.6Å respectively. The specific areas of Fe, Al – Fe, Fe – Cr and Cu – Al montmorillonite are 75 m²/g, 75 m²/g, 68m²/g and 80m²/g, respectively. Element chemical analysis showed that the content of Fe, Fe – Cr, Al – Fe catalysts were 26.93%, 31.03%, 28.20%, respectively. The calcination temperature of PILCs should be lower than 480 °C.

Keywords: Environmental Engineering, Inorganic – Pillared Clay, Preparation, Characterization

1 Introduction

Pillared clays constitute one of the most widely studied families among the new groups of microporous materials developed by molecular engineering. These solids, also known as cross – linked clays or pillared interlayered clays (PILCs), are obtained by exchanging the interlayered cations of layered clays with bulky inorganic polyoxocations, followed by calcination. The intercalated polycations increase the basal spacing of the clays and, upon heating, they are converted to metal oxide clusters by dehydration and dehydroxylation. These metal oxide clusters, named pillars, are inserted between the clay layers, yielding temperaturestable oxide pillars that permanently keep the layers apart, preventing its collapse. As a result, an interlayer space of molecular dimensions, a two – dimensional porous network, is generated. After pillaring, the presence of this new porous structure and the incorporation of new active sites presents several possible applications of these materials^[1-7].

Inorganic pillared montmorillonite possesses not only the unique adsorptivity, dispersivity, porosity, rheological behavior and surface acidity of the clay mineral but also the multivariate functional groups and the reactivity of the organic compound. PILCs have been used as adsorbent in numerous studies. The main utility of this type of solid comes from the fact

that a controlled porosity can be generated by incorporating several elements in between the layers of expandable clay. The newly formed porous material can be systematically regulated by varying the size, the form and the distance between the exchanging cations. Two strategies have been explored in the use of PILC in adsorbent as a support for the active phase by having the active phase forming the pillar itself^[8,9].

In this paper, several typical PILCs, Fe – PILC, Fe – Cr – PILC, Fe – Al – PILC and Cu – Al – PILC were preparation and were characterized by XRD, N₂ – BET and chemical analysis techniques.

2 Methods and materials

The natural bentonite used, which consists mainly of Ca²⁺ – montmorillonite, was obtained from Inner Mongolia, China.

The natural bentonite has a CEC of 108mequiv/100g and the structural formula calculated from chemical analysis and CEC measurements is: Na_{0.017} K_{0.02} Ca_{0.39} [Fe_{0.17} Mg_{0.55} Al_{1.26} Ti_{0.02}]₂ [Si_{3.96} Al_{0.04}]₄ O₁₀ (OH)₂ · 49

H₂O The clay sample was ground and the fraction which can pass a 100 mesh sieve was used in this study (Zhu et al., 2007). The intercalant solution was prepared by titration of an Al³⁺ / Fe³⁺ / Cu⁺ cationic solution with 0.2 mol/L NaOH. The cationic solution contained 0.18 mol/L of FeCl₃ (named Fe –

PILC), 0.18 and 0.02 mol/L of FeCl_3 and CrCl_3 (named Fe - Cr - PILC), 0.18 and 0.02 mol/L of FeCl_3 and AlCl_3 (named Fe - Al - PILC), 0.18 and 0.02 mol/L of AlCl_3 and CuCl (named Cu - Al - PILC), respectively. The NaOH solution was slowly added to the cationic solution at 60°C until the OH/cation molar ratio was equal to 1.9. The intercalant solution was added to the clay suspension under stirring. The final clay ratio was equal to 3.8 mol/g of dry clay. After 24 h, the pillared clay precursor was washed until total elimination of chloride ions, and dried at 60°C . When the PILCS are calcination by CO for 3h (10ml/min)^[12,13].

XRD analysis was performed on oriented samples prepared by spreading of the sample suspension on a glass slide, followed by drying at room temperature. The XRD patterns were obtained with a PW 1130/00/60 Philips diffractometer using Cu K α radiation ($\lambda = 1.5405 \text{ \AA}$). Chemical analysis was carried out on a Perkin - Elmer 3100 spectrometer after dissolution of the sample with several acids (HF, HClO_4 , HCl) for 24 h, and HNO_3 in a second. The surface areas and micropore volumes were determined by the respective N_2 - BET isotherms. SEM analysis was using instruments for the Netherlands PHILIPS - FEI produced XL30ESEM - TMP environmental scanning electron microscope.

3 Results and discussion

3.1 XRD

The XRD and textural data of fresh PILC sorbents are shown in Fig. 1. The chemical compositions of the samples are depicted in Table 1.

From the XRD, all the fresh samples show close values for d_{001} . The XRD of calcination PILC sorbent is reported in Fig. 2. The X - ray diffraction peaks at $2\theta = 24^\circ, 30^\circ$ and 41° exist in the calcination Fe - PILC, Fe - Al - PILC and Fe - Cr - PILC samples, indicating the presence of Fe_3O_4 (JCPDS: 89 - 6466). The X - ray diffraction peaks at $2\theta = 23^\circ, 25^\circ$ and 53° exist in the deoxidized Cu - Al - PILC samples, indicating the presence of CuO (JCPDS: 89 - 5895).

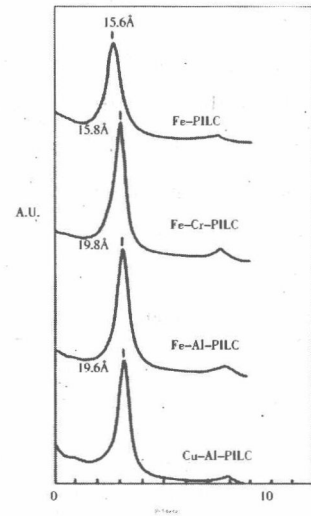


Fig. 1 The XRD patterns for the fresh Fe - PILC, Fe - Al - PILC, Fe - Cr - PILC and Cu - Al - PILC sorbent

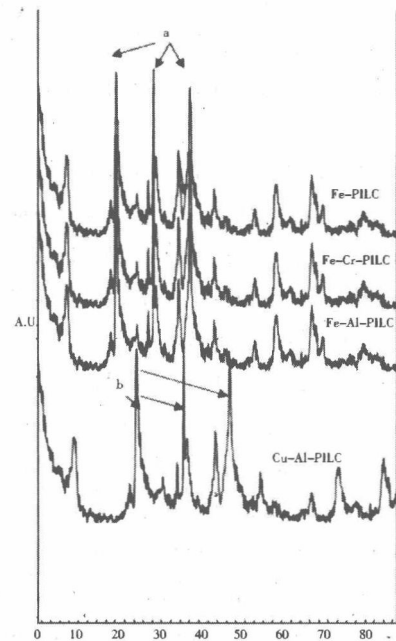


Fig. 2 The XRD patterns of the calcination Fe - PILC, Fe - Al - PILC, Fe - Cr - PILC and Cu - Al - PILC sorbents.

3.2 BET and CA

From the BET, surface areas range 160 ~ 280 m^2/g , with low microporosity and stability up to 500°C . If we take into account the precision of the BET theory used to determine the surface areas (about 10% ~ 15%), one will find that the difference is not large so that different catalytic properties can be relat-

ed to surface chemical properties rather than textural properties.

Fe content in the pillared clay is determined and shown in table 1. In the Fe - PILC, the Fe content is 26.93%. In the Fe - Al - PILC, the Fe content is 31.03%. In the Fe - Cr - PILC, the Fe content is 28.20%.

Table 1 Characterization of the clay samples

Catalysts	d_{001} (Å)	S_{BET} (m ² /g)	Fe (wt %)
Ca - montmorillonite	15.2	56.50	0.50
Fe - PILC	15.6	75.09	26.93
Fe - Cr - PILC	15.8	45.13	31.03
Fe - Al - PILC	19.8	68.10	28.20
Cu - Al - PILC	19.6	79.93	-

3.3 SEM, TG - DTA and EDS

A variety of PILCs are irregular sheet structure, and contour lines are more clearly manifested as cloudy, with a clear sheet structure, and for the thick lamellar structure of a collection of high dispersion. Comparison of four kinds of PILCs adsorbent, Cr accession will be a marked increase in the adsorbent pore structure, size of 0.1 ~ 1 μm, pore size distribution uniformity; Al accession will significantly increase the lamellar structure, and 500 - fold amplification can clearly see that lamellar collection of it.

We can seen through the surface and the face form further amplification, face each other with the end - adsorbed to form a "card room - like" octahedral lamellae structure, which is the original soil with bentonite the same theoretical structure demonstrated that in the modification process, carried out is the ion - exchange, but did not change its original structure. In the "card room - like" side of the existence of irregular 0.1 ~ 1 μm holes, these holes increases the specific surface area for adsorption have a positive effect.

From the Figure 4, the first endothermic peak of Fe - PILC is at 120.32 °C, mainly to get out of the free water and interlayer water, weight loss of about 14.30%, and the second endothermic peak appears in the 485.46 °C, this range is about 4.92 weight loss %, Fe - PILC at this time the octahedral lamellae began to decarboxylation reaction, but the overall

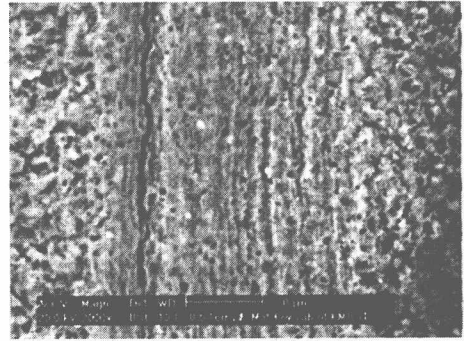


Fig. 3 SEM details of the Fe - PILC

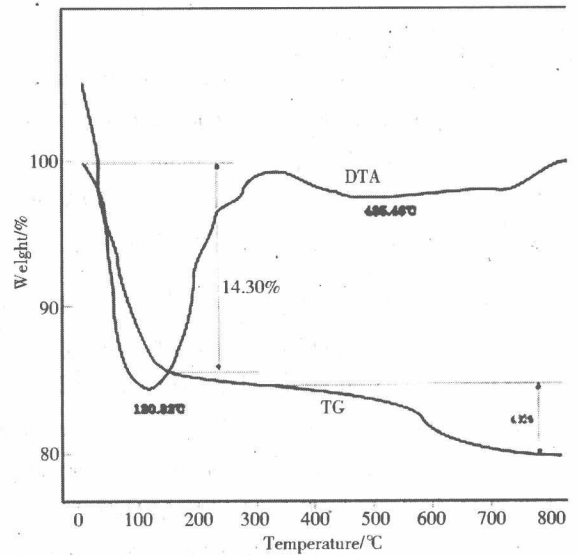


Fig. 4 The TG - DTA patterns of Fe - PILC

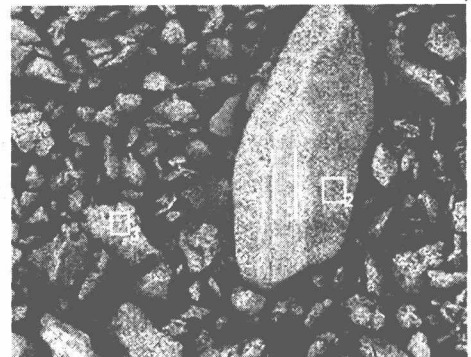


Fig. 5 EDS of the Fe - PILC

layer structure remained in place, the temperature shows, Fe - PILC structural stability. It can be obtained that the calcination temperature of PILCs should be lower than 480 °C.

Table 2 EDS data of the PILCs

Number	Element	Wt%	At%
1	C	12.22	23.15
	O	31.08	44.21
	Mg	01.44	01.34
	Al	04.89	04.12
	Si	16.12	13.06
	Cl	00.30	00.19
	Ti	00.59	00.28
	Cr	01.36	00.60
	Fe	32.01	13.04
2	C	14.51	27.03
	O	30.16	42.19
	Mg	01.49	01.38
	Al	04.62	03.83
	Si	14.25	11.35
	Cl	00.28	00.18
	Ti	00.59	00.28
	Cr	01.50	00.65
	Fe	32.16	12.88
3	C	14.68	24.83
	O	38.54	48.94
	Mg	01.76	01.47
	Al	05.02	03.78
	Si	17.42	12.60
	Cl	00.26	00.15
	Ti	00.47	00.20
	Cr	01.06	00.42
	Fe	20.39	07.42

Since pillared agent, adsorbent array of effective metal content (Fe) significantly increased, while the surface of adsorbent did not detect the metallic elements Fe - based points, which shows Fe metal has entered the bentonite layer, and the different catalyst components in the Si, Mg, K, Mn content is roughly equal, indicating the composition of bentonite and other ingredients have not changed.

4 Conclusions

The XRD shows the d_{001} of Fe, Fe - Cr, Fe - Al and Cu - Al montmorillonite were 15.6Å, 15.8Å, 19.8Å and 19.6Å, respectively. The specific areas of Fe, Al - Fe, Fe - Cr and Cu - Al montmorillonite are 75.09 m²/g, 75.13 m²/g, 68.10m²/g and 79.93m²/g, respectively. Element chemical analysis showed that the content of Fe, Fe - Cr, Al - Fe sorbents were 26.93% , 31.03% , 28.20% , respective-

ly. The calcination temperature of PILCs should be lower than 480 °C.

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Prevention and control of Eutrophication of China's Lakes

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Abstract: With the escalation of Lake Eutrophication, and the frequency of algal bloom outbreaks is getting higher and higher, which is becoming a major environmental bottlenecks in restricting social and economic sustainable development in China. It is urgent to carry out studies on lake nutrient criteria and develop ecoregion nutrient criteria fitted to China's regional characteristics, as theoretic basis for lake eutrophication prevention, control and management. The wide survey should be firstly carried out to get a deep judge of China's lake eutrophication problems and their regional differences. Then, nutrient ecological regions, which are the basic units for the eutrophication standards, need to be determined. Furthermore, nutrient criteria should be developed for each ecoregion, to form the scientific basis of the development of eutrophication standards. Based on ecoregion nutrient criteria, according to different ecoregional nutrients load, different trophic state of lakes and different geographic features, the eutrophication control standard and national lake eutrophication control strategy should be established to direct nutrient total mass control and reduce nutrient load of ecoregion lakes.

Keywords: Lake eutrophication, nutrient criterion, nutrient capacity control

Introduction

There are about 2300 lakes with area of more than 1 km² in China. With the development of industry and agriculture, the speed and scope of lake eutrophication induced by human activities is accelerating. The surveys on eutrophication for 34 Chinese lakes during 1978—1984 indicated eutrophic and hypereutrophic lakes occupied 26.5% of the total number of surveyed lakes. However, the monitoring data for 17 important freshwater lakes and 10 large reservoirs in 2000 (SEPA, 2000) implied eutrophic and hypereutrophic lakes occupied 76.5% of the total number of monitoring lakes and mesotrophic reservoirs occupied 70%. Furthermore, in 2005, eutrophic and hypereutrophic lakes hold 80% of 10 monitoring lakes, 88.9% of 9 large reservoirs were mesotrophic and mesoeutrophic (SEPA, 2005). Nowadays, Eutrophication is one of the major environmental problems of China's lakes and the frequency of algal bloom outbreaks is getting higher and higher, which may result in a nationwide lake eutrophication disaster and hamper social sustainable development.

State and local governments promulgated a series of policies, regulations and management measures to prevent and control lake eutrophication. Scientific research, prevention and control of eutrophication of

lakes were developed from national 7th five year plan in China. Although nutrient standard as total nitrogen and total phosphorus are prescribed in Surface Water Quality Standards of PRC (GB3838-2002), but responding indicators of water ecosystem are not settled (for example, chlorophyll a which characterizes primary productivity, biomass indicators of lake). Moreover, because of diverse geographic, climate conditions and ecosystems, single national nutrient criteria for lakes and reservoirs are not appropriate. Instead, nutrient criteria must be developed at the State, regional or individual water body levels. Compared with the developed countries, in China the lake eutrophication control and management are highly inadequate. Thus, it is urgent to carry out studies on lake nutrient criteria and develop ecoregion nutrient criteria fitted to China's regional characteristics, as theoretic bases for lake eutrophication prevention, control and management. The wide survey should be firstly carried out to get a deep judge of China's lake eutrophication problems and their regional differences. Then, nutrient ecological regions, which are the basic units for the eutrophication standards, need to be determined. Third, nutrient criteria should be setup for each ecoregion, to form the scientific base of the setup of eutrophication standards. Based on ecoregion nutrient criteria, according to different ecoregional nutrients

load, different trophic state of lakes and different geographic features, the eutrophication control standard and national lake eutrophication control strategy should be established to direct nutrient total mass control and reduce nutrient load of ecoregion lakes.

1 Lake database and nutrient ecological divisions

In China, the significant regional characteristics of lakes, historical data on lakes are lacking and may be insufficient to identify lake classes and reference condition. The development of nationwide regional nutrient criteria and eutrophication control strategy requires the availability of an extensive amount of data from across the country for analysis and evaluation. Data may come from existing sources or can be collected from new survey. Nutrient - related data for lakes have the potential to provide the basis for the nutrient ecological divisions and development of nutrient criteria on a regional level. Moreover, the lakes' characteristics and their regional differences must be investigated, establish dynamic basic database of China's lake basin, and fully predominate characteristics of China's lake basin and the level of nutritional status. Further studies will be focused on the driving forces and their regional differences of eutrophication in China's lakes, to find out how natural conditions and human activities (especially human activities) impact lakes' trophic states, and how lake ecosystem responds to nutrients input and accumulation. Both statistical analysis methods and modeling techniques will be used, as well as data mining and data finding techniques, expecting to make full use of data collected and to get a well understand of China's lake eutrophication problems.

The process of identifying geographic divisions (i. e., regionalization) is part of a hierarchical classification procedure with the purpose of grouping similar lakes together. Expectations can be developed for nutrient concentrations and loadings in each of the regions and criteria derived from those expectations (USEPA, 2000).

On the basis of survey of the regional differences and its distribution rules of eutrophic lakes across the

country, to find out completely the characteristics of the structure and function of lake ecological system in China, with synthetically use of modern statistics, landscape element analysis, "3S" and other technologies and methods, to interpret the spatial coincidence in all geographic factors that cause or reflect differences in lake ecosystem patterns. These factors include geology, physiography, vegetation, climate, soils, land use, wildlife, nutrients, and hydrology. There are general similarities in the quality and types of ecosystems as well as in natural and anthropogenic characteristics that affect nutrients in same nutrient region.

The general approach to the lake nutrient regionalization process is to establish divisions at the broadest level and then to continue to stratify to a reasonable point. A three - lever regionalization system for the national scale should be developed to provide a framework for developing nutrient criteria in China. The nutrient regions can form the basis for initial development of nutrient criteria.

2 Establish ecoregion nutrient criterion

Based on the investigation of regional difference and nutrient ecological division, the indicators of nutrient criteria in typical nutrient ecoregion will be selected after identified the relations of candidate variables for different lake types. Nutrient variables involve necessarily the indicators of overenrichment and lake geographic differences, and possess the characteristics of relatively stability, ecoregion and eutrophication prediction. Establishing lake reference conditions for each of the physical lake classes within each ecoregion or subdivision of that region is a critical part of the nutrient criteria development process. Reference conditions for each of these classes are the quantitative descriptions of lake conditions used as a standard for comparison purposes (USEPA, 2000).

Reference conditions will be established through several methods of reference condition establishment applying to different nutrient region, such as as statistical distributions of direct observation of reference lakes, model prediction and extrapolation, and paleolimnological reconstruction (Huo et al., 2009).

Through analyzing historical and existing water quality data and biological information, statistical approach could ensure the established criteria is applicable for most lakes (Zheng et al., 2009). However, the reference conditions are not specifically established by this approach when the lakes lack observation or significantly impacted by human activity. Model prediction and extrapolation can be used for lakes suffered anthropogenic disturbances, and needs to be recalibrated and tested with large data sets (USEPA, 2000; Solheim, 2005; Dodds et al., 2006).

Paleolimnological reconstruction approach tracks the natural background concentrations of lake nutrients and quantitatively restores past conditions by using sediment cores, and requires complex analyses and expert judgments (USEPA, 2000; Yang et al., 2008). It is difficult to reconstruct the past nutrient condition of shallow lakes in which sediments are easily disturbed. Most of lakes have been impacted seriously by human activity in China, lack historical data record, and have significant geographic differences. Finally, reasonable numeric nutrient criteria will be developed by considering designated uses, antidegradation policy, endangered species, and downstream effects.

3 Establish eutrophication control standards

Lake nutrient ecological regions are the basic control unit of lake eutrophication. The eutrophication control standards of different ecoregion should be developed based on relationship between ecoregion nutrient criterion and lake eutrophication control standards. Considering effectively controlling eutrophication and the combination of aquatic ecosystem, human health, function of lakes, social economic conditions and environmental management target, evaluation index system of lake eutrophication should be established and control standard value of lake eutrophication indicators should be scientifically identified for different ecoregions. Furthermore, lake eutrophication control standards and classification system of different ecoregion would be developed. In addition, it is necessary to evaluate the technical feasibility, economic rationality, social adaptability of lake eutrophication

control standards, which provide support for continuous lake nutrient total mass control, reduced systems and ecological restoration.

4 Establishment of total nutrient mass control and reduction system

Based on nutrient production, transformation and geographical differences of different ecoregion eutrophication, total capacity control strategy can be established including integrated control of point and non-point sources. Different pollution sources should be listed to achieve distribution of pollutant loads; and setup critical safety point to insure improvement of water body quality target. The maximum allowable nutrients loads should be calculated basing on the function of different lake integrated water body and self-purification capability of water body. Total capacity of water body should consider the factor of safety and seasonal variation, and more suitable for ecological safety and resource protection of the new economic development. The objectives and tasks of water environmental protection need to be brought into local governments and social development plan. Nutrient reduction plan and guidance programs should be developed according to the characteristics of lake watershed, so that provide basis for decision-making of lake eutrophication control and integrated management in each region.

5 Conclusion

There are thousands of lakes in China, distributing in different parts of China, and most of which are facing serious eutrophication problems as a result of fast economic development and population growth. As natural conditions and human activities differ a lot in different parts of China, eutrophication problems may have great differences in lakes. Thus, a single-value standard (GB3838-2002) couldn't satisfy the requirements of lake management and eutrophication control.

Eutrophication ecoregional standard should be established for China's lakes, aiming to provide scientific guidance in lake management and eutrophication control. Thus, a wide investigation should be

firstly carried out to get a deep understand of China's lake eutrophication problems and their regional differences. Nutrient ecological region, which is the basic units for the eutrophication control, need to be determined. Nutrient criteria should be developed for each eco-region, to form the scientific base of the setup of eutrophication standards. Furthermore, the eutrophication standards could be established, and related managing instruments should also be setup, in order to ensure the standards be effectively carried out.

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水力空化技术灭藻

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摘要:应用一套水力空化装置对富营养化水体中的铜绿微囊藻类进行了灭杀研究,结果表明水力空化对铜绿微囊藻具有一定的灭杀效果。2.5 L浓度为 $3.5 \times 10^6 \text{ mL}^{-1}$ 的铜绿微囊藻在25℃的条件下经过20 min的水力空化灭杀,在培养7 d时发现:使用孔径为6 mm的单孔板孔板,将其安装在水泵吸入口,杀藻率可达到41%,而将孔径为6 mm的单孔板串联9孔多孔板(单孔孔径为2 mm)安装在水泵吸入口,其杀藻率可达到58%,水力空化还可以强化 H_2O_2 的杀藻作用,单独使用 $1 \text{ mmol} \cdot \text{L}^{-1}$ 的 H_2O_2 进行杀藻时,其杀藻率仅为29%,而当与水力空化联合杀藻时,其杀藻率可达65%。

关键词:水力空化,杀藻

Removal of Algae by Hydrodynamic Cavitation Technology Weimin Wang¹, Zhilin Wu^{1,2,*}, Yongchun Zhang¹, Yimin Zhang¹, and Bernd Ondruschka² (1. Nanjing Institute of Environmental Science, MEP of China, Jiangwangmiaostr. 8, 210042 Nanjing, China; 2. Institute of Technical Chemistry and Environmental Chemistry, Friedrich Schiller University of Jena, Lessingstr. 12, D-07745 Jena, Germany)

Abstract: A bench scale device for creating hydrodynamic cavitation has been set up. In a typical run, 2.5 L of $3.5 \times 10^6 \text{ mL}^{-1}$ algae solution (*Microcystis aeruginosa*) was circulated at 25℃. The highest removal rate (41% after 7 days cultivation) appeared by the treatment using 6 mm single-hole plate at the suction site of the pump in 20 min. Under the combination of the single-hole (6 mm) plate at 1# position and the 9-hole (2 mm) plate at 3# position, the removal rate can achieve 58% by suction treatment in 20 min and after 7 days cultivation. The removal rate reached only 29% in 7 days cultivation after the oxidation of $1 \text{ mmol} \cdot \text{L}^{-1} \text{ H}_2\text{O}_2$ alone for 20 min, but the oxidation-removal rate was obviously enhanced to 65% by hydrodynamic cavitation.

Keywords: Hydrodynamic Cavitation, Algae Removal

1 前言

空化是气泡在液体中产生、生长和溃灭的过程^[1],空泡在溃灭时可形成伴随有极端高温高压的物理及化学变化^[2]。水力空化是空化的一种,近来在废水处理^[3-6]和水消毒^[7]领域得到应用。长期以来,关于杀藻的研究受到了国内外学者的高度重视。传统的藻类抑制(灭杀)方法有物理法、化学法以及生物法^[8]。但因各种方法都有其局限性,至今仍然没有一种既可抑制(灭杀)藻类生长又不造成二次污染的杀藻方法在实际中得到应用。本研究试图以水力空化技术对藻类进行灭杀,以期在这方面做一些新的探索。

水力空化是指当液体内部局部压力降低时(主要是由高流速引起压力降低),液体内部或液固交界面上蒸气或气体空穴的形成、发展和溃灭过程^[1]。人们最初对水力空化的研究是为了避免和减轻其对水利设施、水利机械等带来的危害。随着对水力空化机理和现象研究的深入,人们逐渐认识

到,利用水力空化瞬间产生的巨大能量可以实现对一些物理和化学过程的强化。

2 实验部分

2.1 理论基础

水力空化可通过使流体速度发生变化引起压力变化而产生。流体流过一个收缩装置(如孔板)时会产生压降,若液体中某处的压强达到或低于某一临界值(临界压强),在温度不变的情况下,原来溶解在流体中的气体会释放出来,流体汽化而产生大量空泡并迅速膨胀,空泡在随流体进一步流动的过程中,周围的压力逐渐恢复,空泡体积将急剧缩小直至溃灭。空泡形成、膨胀、收缩以及溃灭的过程称为空化现象。

研究表明:空泡溃灭时会产生瞬时局部高温(约5200 K)、高压(50 MPa以上),并能形成强烈的冲击波和速度高达 $100 \text{ m} \cdot \text{s}^{-1}$ 以上的微射流且

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