

## TREATMENT OF DOMESTIC WASTEWATER USING UP-FLOW ANAEROBIC FILTER

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**Abstract.** This paper presents results of the lab-scale anaerobic filter and pilot-scale units of anaerobic baffled filter used for municipal wastewater treatment by low temperature. The lab-scale reactor had a volume of 1.7 dm<sup>3</sup>, being operated at hydraulic retention time (HRT) of 12 h and 24 h. The wastewater temperature was adjusted at 23 °C, 15 °C, and 9°C, respectively. After almost one year of continuous monitoring, the lab-scale upflow anaerobic filter system produced very good results in terms of COD and BOD<sub>5</sub> removal, and also very low solids concentration in the final effluent. The average results of COD and BOD<sub>5</sub> removal varied from 41 to 95 % in dependence on temperature and values of hydraulic retention time. The pilot-scale experiments were realized in anaerobic baffled filter with total volume of 2 m<sup>3</sup>. This reactor was installed on wastewater treatment plant (WWTP) Bratislava-Petrzalka and was fed with raw wastewater under HRT of 24 h. The start-up and operation of anaerobic filter was executed under winter climate condition. The observed COD and BOD<sub>5</sub> removal efficiency was in average 66 % and 68 %, respectively. Very high efficiency was achieved for suspended solids removal - 95 % left only.

**Keyword.** Suspended solids; domestic wastewater; an aerobic.

### INTRODUCTION

Anaerobic processes have been used for the treatment of concentrated municipal and industrial wastewater for well over a century [1]. With the progress in engineering and developments in chemistry, biochemistry, and microbiology [3], anaerobic treatment has been gaining increasing momentum as the main means of organic matter removal in wastewater. Especially in high-strength organic wastes treatment [6], anaerobic systems are superior to conventional aerobic systems in saving capacity and operational costs, reducing production of sludge and producing of biogas [8]. Anaerobic filters (AFs) were first described in 1969 by Young and McCarty, and have been studied over the past 40 years [10]. AF is a fixed bed fixed film anaerobic reactor, in which porous media are used for immobilization of microorganisms [12]. This type of reactor has been widely used due to its large biomass attachment, high organic loadings, long sludge retention time, easily restarting and simple operation. When wastewater flows through the reactor, suspended solids (SS) in wastewater can be filtrated directly by the submerged support media [13], hence additional filter device will be unnecessary. However, there are still some disadvantages for practical application of an aerobic filter, such as media clogging [9], long start-up period and temperature limitation. Therefore, improving these weak points is one of the most significant aspects in an aerobic wastewater treatment. To achieve better treatment performance, filter media is an indispensable contributor for anaerobic filter, because it facilitates the retention of biomass in the reactor that is longer duration achieving longer mean cell residence time [21]. In AFs, the biomass retention is accomplished by fixing microorganisms in the form of biofilm attached on the media surfaces and suspended biomass trapped within the spaces of filter media. Previous studies have indicated that support material markedly affected the rate of attachment and growth of bacteria converting acetic acid to methane, and have also proved that media surface texture and porosity played a significant role in the performance of up-flow anaerobic filters [12]. Therefore, selecting the optimum biofilm support media is seemed to be of great importance in AF treatment. It has been reported that many materials with high media porosity have been applied to support the biomass in the form of microbial films, including special ceramic, polyurethane foam, PVC, zeolite, glass, carbon filter and soon [4]. In this paper, a novel ceramic particle from local

clay in Xuzhou (Jiangsu Province, China) has been studied in our laboratory. Some characteristics of CCPs were analyzed to evaluate the performance of synthetic wastewater treatment, including bulk density, grain density and water absorption. Some other characterization methods including X-ray powder diffraction (XRD) and scanning electron microscope (SEM) were also carried out for further analysis. According to the experimental results, CCPs have presented higher total porosity and roughness, meanwhile lower bulk and grain density compared to sludge-fly ash ceramic particles (SFCP), which was studied by [11], and have reached the index requirements of media filler. Sequentially, after studying the potential use of novel filter media for wastewater treatment in details, the AF was chosen to investigate the influence of different media height and organic loadings for ascertaining the operation of AF and the applicability of CCPs for their further industrialization. In addition, long start-up period and temperature limitation are always the weak points in AF treatment, and so far few research papers have investigated anaerobic bio-filter under a relatively lower temperature (16–21 °C) all over the world. Therefore, the main objective of this paper was to investigate whether the new type of ceramics employed in self-designed anaerobic filter have superiority compared to other fillers during start-up phase and stable stage in ambient temperature, especially in relatively lower region.

## METHODOLOGY

### Reactor

As indicated in Fig. 1, the lab-scale cylindrical reactor made from polymethyl methacrylate had a diameter of 200 mm with the height of 1.7 m. The column was packed with novel filter media (CCPs) for 1 m high, and the effective volume for biochemical action was 15 L. On the top of packing layers, some gravel was added as barriers to prevent CCPs from floating out. In addition, graded gravel layer with the height of 10 cm was settled at the bottom of the filler layer as filter supporting bed. Influent was fed into the column from the bottom of the reactor with a variable speed peristaltic pump. The injected wastewater flowed through the buffer zone and supporting board, and evenly passed through the new CCPs filter bed. The treated effluent was discharged from the top of the reactor by outlet pipe connected to an effluent tank. Six sampling ports were placed at 30, 50, 70, 90, 110, and 130 cm from the bottom flange. Periodic backwashing was necessary in order to prevent clogging of fillers. The system was operated at ambient temperature ranging from 15 to 31 °C [7].

### Synthetic wastewater

At the beginning of the experimental period, glucose ( $C_6H_{12}O_6$ ), ammonium sulfate ( $(NH_4)_2SO_4$ ) and potassium bi phosphate ( $KH_2PO_4$ ) were used to prepare the main parts of the synthetic wastewater, meanwhile sodium bicarbonate ( $NaHCO_3$ ) was dosed to adjust pH. Then in order to complicate the carbon source and reduce the intake of sulfate radical, another synthetic wastewater made up by sucrose ( $C_{12}H_{22}O_{11}$ ), ammonium chloride ( $NH_4Cl$ ) and potassium bi phosphate ( $KH_2PO_4$ ) was used to replace the original synthetic wastewater gradually after 2 weeks of experimental period.

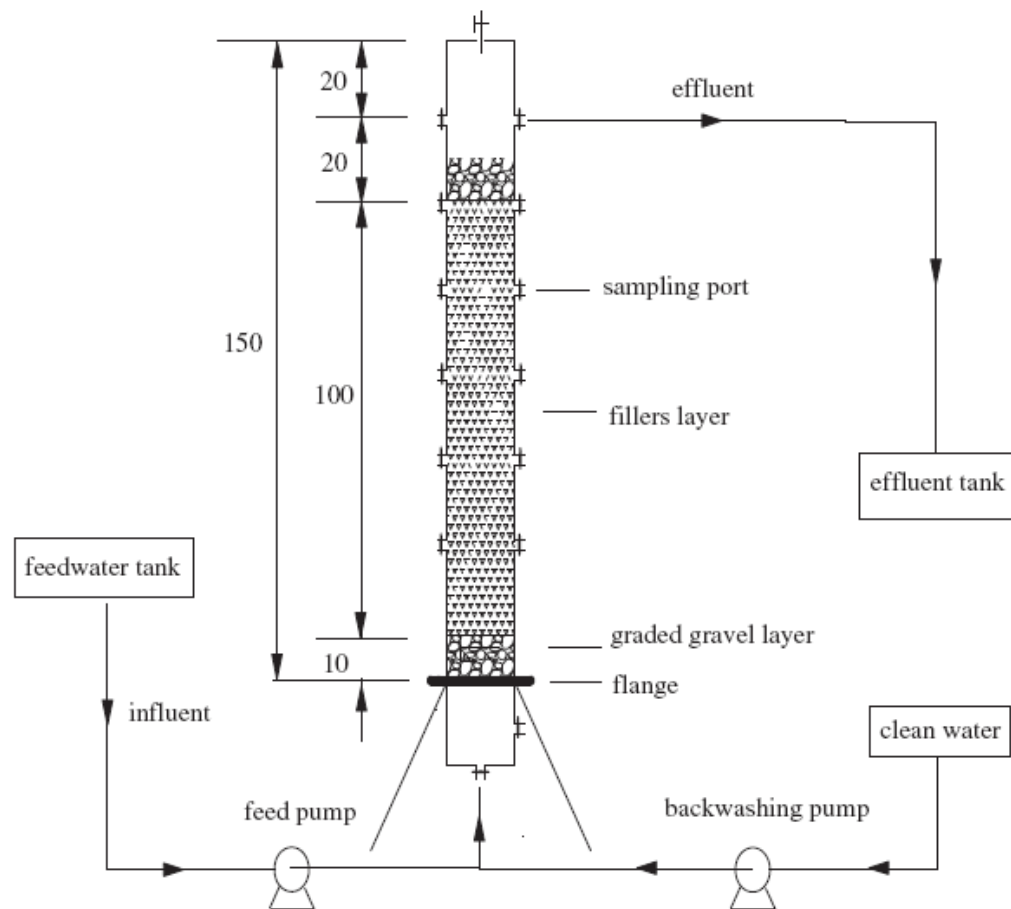


Figure 1. Schematic diagram of experimental set-up (dimensioning unit: cm).

At the same time, essential nutrients to microorganisms including  $\text{Co}^{2+}$ ,  $\text{H}_3\text{BO}_3$ ,  $\text{Fe}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Mo}^{6+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Se}^{4+}$ ,  $\text{Al}^{3+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$  were added. Ethylene Diamine Tetraacetic Acid (EDTA) and sodium bicarbonate ( $\text{NaHCO}_3$ ) were used as the buffer substance. As for the proportion of substrate nutrient, the ratio of C:N:P was controlled at 100:5:1 and (200 to 300):5:1 during the start-up period and steady state, respectively.

Table 1. Chemical components of clay (%).

	$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	$\text{CaO}$	$\text{K}_2\text{O}$	$\text{Fe}_2\text{O}_3$	$\text{TiO}_2$	$\text{MgO}$	$\text{Na}_2\text{O}$
Clay	70.53	16.59	1.33	2.29	6.43	0.75	0.99	1.10

#### Inoculation and start-up

AF system was inoculated with concentrated activated sludge, which was obtained from a digestion basin of Jinan Wastewater Treatment Plants, and synthetic wastewater of equal volume was filled in the reactor as nutrient supply. During the first 2 weeks, the mixture was reserved in the reactor for a few days under an aerobic condition, and then synthetic wastewater was continually fed into the reactor at a low flow rate. To stimulate the growth of microorganism and improve the buffering capacity of the reactor, higher nitrogen content would be appropriate during start-up period, thus the ratio of C:N:P

was selected as 100:5:1. Basic operation parameters in the acclimatization period were as follows: hydraulic retention time (HRT) of 24 h, total flow rate of 0.83 L/h, COD concentration of 1000 mg/L and organic loading rate (OLR) of 1.3333 kg COD m<sup>3</sup> /d. An appropriately backwashing time of five days was chosen for accumulating biomass.

### Analysis

#### Analytical methods

During the experiments, both influent and effluent were collected, and all the samples were stored at 4 °C for less than 24 h before measurement. Various parameters, including pH, COD and ammonia nitrogen (NH<sub>4</sub>-N), were determined according to the monitoring and analysis methods of water and wastewater [8]: potassium dichromate method and colorimetric method were used for COD and NH<sub>4</sub>-N analysis respectively. The measurements of every item for each sample were repeated for three times, and the average value was selected for determination.

#### Characterization of the CCPs

CCPs were produced from common clay and straw was utilized as expansion agent. All the ceramists were approximately ellipsoidal shaped, and size ranged from 10.0 to 20.0 mm. The chemical components of clay were determined by energy dispersive X-ray fluorescence spectrometer (EDX) and the results are listed in Table 2. As shown in Table 2, some essential physical properties of filter materials were analyzed, such as bulk density (which included all voids and spaces in the volume), grain density (also called particle density, showed the apparent specific gravity of the support

**Table 2. Physical properties of CCP.**

	Bulk density (kg m <sup>-3</sup> )	Grain density (kg m <sup>-3</sup> )	Water absorption (%)	Voidage (%)	Particle diameter (mm)
Product (CCP)	434.6	812.6	8.235	53.48	10.0–20.0
GB/T17431-1998	<500.00		<15.00		

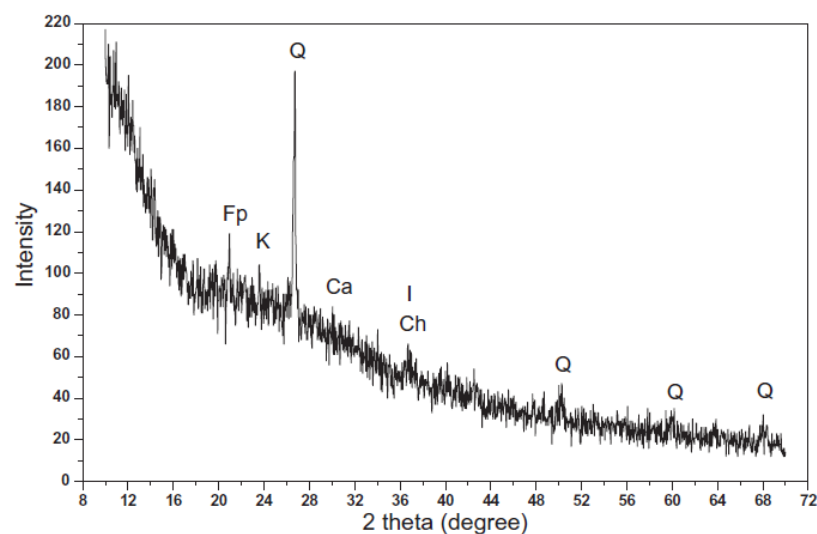
Including all intra particle voids), voidage ((1-bulk density/grain density) × 100%) and water absorption (characterized with weight difference between the dried and water saturated samples) [5]. Before the tests, the samples were settled into an oven (105 °C) for 4 h [12], then cooled down to the room temperature (25 °C). In order to study the properties of carrier bodies before and after using in bio filters, a Hitachi S-520 scanning electron microscope (SEM) was used to observe the surface and the cross section of CCPs. As for the clay ceramists attached with bio film, different samples were individually taken out from the bio-filters from different heights, and treated with glutaraldehyde 2.5% solution to fix the cells, then followed by dehydration in ethanol, replacement with isoamyl acetate and freeze-drying. Thereafter the procedure was carried out in the same way as the unused ceramists [14].

## RESULTS AND DISCUSSION

#### Pore characteristics and morphology analyses

Rough surface and porous structure are essential conditions to the successful microbial growth. Suppl. Figure (A–C) indicates the morphology of CCPs before and after the inoculation operation. As shown in Suppl. Fig. A that the surface of CCPs was rough and with many small pores, which achieved large specific surface area. Rough surface and large specific surface area are two of the important factors that influence the attachment and fixing of microorganism. Moreover, microbes attaching and fixing on the surface of filter media is the key process in bio film formation, which could directly affect the bio film in its organisms as well as physiological efficacy, meanwhile start-up and operation cycle of bio filter. As for Suppl. Fig. B, it could be seen that the cross-section of CCPs was porous and with relatively large pores. Reactor with larger media porosity can obtain high removal efficiency attributable to higher growth of suspended biomass, which has been proved by Show and Tay (1999). Suitable porosity guarantees that the wastewater can flow evenly, and provides microorganism the

appropriate growth environment. These rough surface and porous properties of CCPs were related to their chemical constituents. Generally, ceramic particles contain three kinds of chemical compositions [23]: (1) glassy materials, such as  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ , are major components of ceramics, forming the ceramic framework; (2) gasogenic constituents [21], which generate gaseous bubbles bloating the body of ceramics included carbon, ferric oxide, ferrous oxide and magnetic iron; (3) fluxoxides [20], including  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{MnO}$ , lower the melting point of the glassy phase. Based on the data in Table 1, gasogenic constituents from it made the surface of CCPs rough. Furthermore, fluxoxides made CCPs lower bulk and apparent density, creating a porous structure. After inoculation (observed from Suppl. Fig.C), pores on the surface of CCPs were filled with microbes, which suggested that a suitable amount of microorganisms were immobilized onto the inside and outside surfaces of the pores in CCPs. Based on the analysis of Suppl. Figure (A–C), CCPs presented remarkable capacity for bio-mass attachment, which could be a strong evidence to show that CCPs was an appropriate media for anaerobic filters.



**Figure 2.** XRD patterns of CCP. Q: quartz; K: kaolinite; Fp: feldspar; I: illite; Ch: chlorite; Ca: calcite.

CCPs sampled from different height of the anaerobic filter were examined to observe the microbial distribution according to the method described in Section 2.4.2. Suppl. Fig.C shows the growth of bio film in the CCPs after utilization. In the range between 10 and 50 cm from the bottom flange, fulvous attached -growth flocculent mass could be seen on CCPs, with the average thickness of 2 mm through visual observation. Moreover, through SEM image, it could also be seen from Suppl. Fig.C that the cells were directly attached to the surface of CCPs and bio film was apparently spread the surface of CCPs, with the relatively well structure maintained. However, during the upper height of the filter media, about 50–100 cm, bio film was hardly found no matter through visual observation or SEM image. It can be explained by the fact that in the UAF, microorganisms at the bottom zone grow well and the attached biomass is in a large quantity. Along the media height with the shortage of nutrients in the upper section, it is not enough for bacteria to proliferate, hence the biomass is small [19]. In addition, no clogging or disintegration phenomenon was found, which indicated that reasonable operation made the anaerobic system function well and CCPs were proved to be appropriate as filter media for microorganism growing. Furthermore, lightweight (bulk density of  $434.6 \text{ kg m}^{-3}$  and particle density of  $812.6 \text{ kg m}^{-3}$ ) CCPs were favorable for wastewater flowing evenly due to the large porosity, and the regular shape of ellipsoid greatly reduced the flow resistance. As a result, clogging avoided could attribute to the distinctive structure of CCPs.

Analysis of XRD and trace elements in leachate of CCPs

XRD is widely used in clay mineralogy research. Mineralogical analyses of CCPs carried out with XRD are shown in Fig. 3. It could be noted that CCPs samples have their major peaks assigned to quartz (Q), kaolinite (K) and feldspar (Fp). Minor peaks assigned to illite (I). Chlorite (Ch) and calcite (Ca) were also found in the samples. The mineral composition of CCPs refers to the relative abundance and identity of the clay minerals present in a clay material [18]. However, raw clay materials were sintered under high temperature, and ceramsite samples were formed in this way, thus few differences were presented between the XRD pattern of original clay materials and CCPs.

Clay and clay minerals have been widely used as the main raw materials in different ceramic products for construction materials due to their special properties before and after sintering [16]. Lixiviation test was carried out to know more about the trace elements and the potential toxicity of CCPs. Table 3 indicates some metal elements in the lixivium of clay ceramists, which are treated in the mixed strong acid and shaken for 20h according to HJ/T299-2007 [17]. The result revealed that trace elements (such as Al, Ca, Co, Cu, Fe, K, Mg, Mn, Na, Zn) in lixivium of CCPs were in rich content and potentially favorable to microbe growth. In addition, only few noxious matters were found in the lixivium, with the contents much lower than the limits of the national standards determined by GB5085.3-2007, China (Hazardous Wastes Distinction Standard-Leaching Toxicity Distinction). Therefore, according to the analysis of the two aspects, it could come to the conclusion that this new CCPs was completely suitable as filter media for wastewater treatment.

Superiority of applying CCPs in the anaerobic bio-filter

Performance during the start-up period Simple in operation does not mean that there is no trouble in high rate process. Reactor startup is one of such initial obstacles

**Table 3. Contents of metal elements in lixivium of CCP.**

Metal elements	Content in lixivium (mg L <sup>-1</sup> )	Threshold (mg L <sup>-1</sup> )
Al	0.0602	NA
Ca	12.27	NA
Co	0.002	NA
Cr	0.0183	15
Cu	0.0537	100
Fe	0.0543	NA
K	3.901	NA
Mg	1.965	NA
Mn	0.0268	NA
Na	3.545	NA
Pb	0.0347	5
Zn	0.045	100

Which must be overcome to attain successful process operation [6]. Start-up usually takes about 2–3 months or even up to 3–6 months after biomass development and accumulation rate [8]. The start-up period was carried out at the temperature 29±2 °C. During the first week of the start-up period, the reactor was fed with synthetic wastewater mentioned in Section 2.2, and only pH was measured every day. Sodium bicarbonate was added to guarantee the pH was maintained in the range of 6.8–7.2. Then in the following days, COD and NH<sub>4</sub>-N were also analyzed every other day. The initial COD concentration of influent was 1000 mg/L, and the organic loading rate was controlled to be 1.3333 kg COD m<sup>3</sup> /d. COD removal rate and pH during the start-up period are presented in Fig. 3A. It could be seen that during the initial 2 weeks of the startup stage, pH and COD removal rate fluctuated a lot, with a relatively low COD removal efficiency ranged between 40% and 50%. The reason might be that during initial stages of reactor startup, bio film was not yet formed and the relatively low COD removal efficiency was made by a low concentration of suspended microorganisms from inoculated activated sludge. Then suspended bio-mass grew and gradually developed into bio film. This process is sensitive, unstable and inefficient for organic removal, which can be related to the fact that there exists a variety of complex relationships of microbial flora in aerobic bio film systems. In other words,



with the buildup of various symbiotic relationships amongst the attached microflora, the bio film infrastructure is

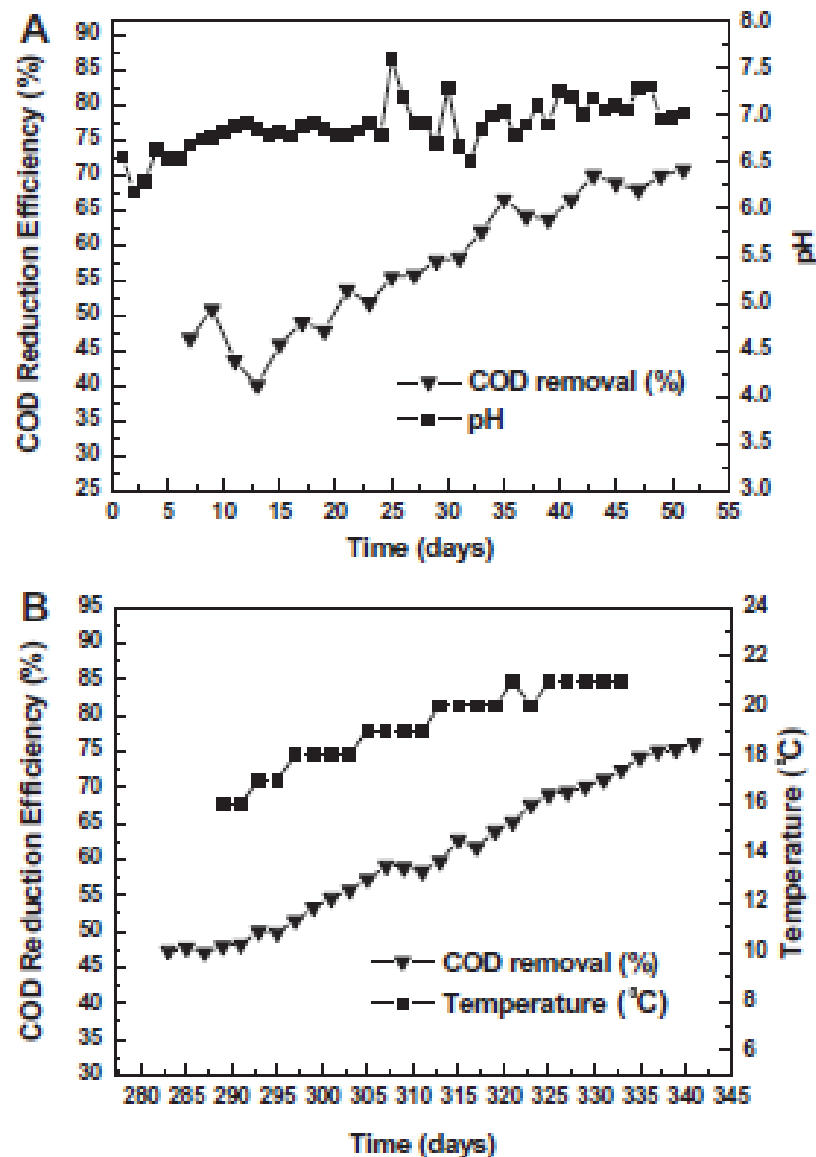


Figure 3. COD removals for different conditions: (A) COD and pH monitoring during the start-up period; (B) COD removal in the relatively lower temperature

Continuously evolving. The anaerobic treatment process is actually a series of biochemical coupling reaction made by these microorganisms, among which the relationship between non methane - producing bacteria and methane -producing bacteria is the most important. For one thing, they are interdependent and symbiotic; for another thing, they also restrict each other, in order to keep a state of equilibrium in this anaerobic system. Thereafter, as waste- water continually flowed through the filter media appropriately, microorganisms kept growing and gradually formed bio film which attached to the clay ceramists. During these stages, influent COD concentration combined with hydrodynamic conditions could properly wash out the dispersed biomass and retain heavier fraction biomass. In the following days, pH tended to be stable, and COD removal increased gradually. It should be noted that between 25th day and 30th day, great temperature fluctuation resulted in the once more wave of pH. However, because of a suitable habitat place with strong heat preservation and buffering capacity provided by the filter media, COD removal rate was not influenced seriously. Then after 40 days, as depicted in Fig. 3A, COD removal and pH both achieved stable state. The

symbols of bio film getting mature gradually were that the ecological system made up by bacteria and various microorganisms achieved the balanced and stable state, which was also directly reflected in the degradation rate of organic matter in the effluent. As a result, the start of this anaerobic reactor, which was only 45 days, greatly reduced the start-up time comparing with the conventional process. All above were attributed to the chemical composition of clay and the structure of CCPs. On one hand, clay contained abundant of trace elements which were essential for microorganic growth. Trace elements in lixivium can stimulate bacterial growth and enhance biomass accumulation in the reactor [1]. On the other hand, the special structure of CCPs, such as surface roughness and piousness, seemed to be the important media characteristic which influenced the bio film formation. It reflects in promoting higher growth of attached bio film and suspended biomass, respectively [9]. Moreover, it has been observed that initial microbial attachment invariably occurred in the crevices of support media. Clay ceramists used in this research had been found to have these two advantages, hence they contributed to a short start-up period with 45 days

## CONCLUSION

Investigation of the novel filter media-CCPs employed in a lab- scale UAF for synthetic wastewater treatment was carried out in this paper. The following conclusions can be drawn: (1) The feasibility of CCPs as filter media by UAFs was demonstrated according to the properties of CCPs. (2) The advantages CCPs brought for UAF were in the following two aspects: shortened the start-up time to 45 days and improved the organic removal rate to 76% at relatively low temperatures. (3) This lab-scale UAF showed an acceptable performance in COD reduction, with the potential for higher COD concentration treatment and actual wastewater.

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