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Remediation of Refractory Organic Matter in Coal-to-Gas Industrial Wastewater Using Coal Ash-Based Compound

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Abstract: As the coal to gas industrial wastewater has high organic concentration that is difficult to degrade, the application research of coal chemical solid waste compounds to the removal of refractory organic wastewater pollutants from coal gasification was studied. According to the characteristics of solid waste compounds, the best combination conditions and the best removal effect of refractory organic matter were determined by single factor experiment, and the possibility of its combination with coagulant PAC was explored. It is found that the removal efficiency of refractory organic matter in coal-to-gas industrial wastewater can be improved, the dosage of PAC can be reduced, and the waste water treatment cost can be saved. Fx composite has good removal effect of COD, turbidity and the refractory organic compounds- total phenols of organic wastewater. When the pollutants in organic wastewater are treated by Fx alone, the best removal rate of COD, total phenol and turbidity reaches 60%, 50.2% and 97% respectively. This has a good carbon emission reduction effect, that contributes to the realization of the low-carbon green cycle development concept of "treating waste with waste".

Keywords: Coal to Gas Industrial Wastewater, Solid Waste Compounds, Refractory Organic Matter, Treatment Cost, Treating Waste with Waste

1. Introduction

“Treating waste with waste” is a green coordinated circular development concept which follows the principle of "reduction, reuse and resource recovery". Developing circular economy is a major strategy of our country, promoting the establishment of a resource-recycling industrial system and a recycling system for waste materials which is of great significance to promote the realization of "carbon peak and carbon neutral" and promotes the construction of ecological civilization, that was proposed by Circular Economy Development Plan in the 14th Five-year Plan.

Coal chemical industry, as the main body of China's long-term energy consumption, occupies an important strategic position in China's energy system [1]. Modern coal chemical industry is mainly based on coal gasification to produce natural gas and methanol, ethylene, propylene and other important petrochemical products [2]. The main direction of clean and efficient utilization of coal in China [3] is to achieve the higher conversion efficiency and the greater product added value. However, modern coal chemical technology still faces key technical problems such as high energy consumption, high water consumption and high emissions [4]. A great deal of waste water, waste gas and solid waste produced in the process of coal gasification bring great pressure to the ecological environment, the "three wastes" treatment technology. Especially the comprehensive recycling technology of solid wastes, is in urgent need of improvement [5].

The general solid wastes produced in coal chemical industry mainly include gasification slag, thermoelectric slag and fly ash.

At present, the reuse of slag and fly ash mainly include [6-8]: 1) making adsorption material [9, 10], Slag and fly ash have loose porous structure, large specific surface area, and contain unburned carbon, silicon, aluminum, iron and other active elements, with good physical adsorption, chemical adsorption and ion exchange adsorption capacity, can be used for adsorption and removal of wastewater pollutants. Yichen Wang et al. modified fly ash with sulfuric acid and applied it to the advanced treatment of coking wastewater [11]. The results showed that the removal rates of COD and turbidity of wastewater by modified fly ash reached 77.7% and 96.7% respectively. 2) making building materials [12], slag and fly ash have similar composition characteristics with cement, concrete and other building materials, and can be used to make cement, concrete, masonry materials, insulation materials and so on. 3) Aluminium recovery [13, 14], in China, bauxite resources are insufficient and import dependence is high. However, slag and fly ash contain high content of alumina, which can be directly used to extract alumina and regenerate aluminum resources. 4) Production of zeolite, catalyst and other high value-added products [15, 16], slag and fly ash are rich in active components such as silicon oxide and alumina, which have good catalytic activity and are also good precursors for the preparation of zeolite.

The general solid waste landfill of a coal-to-gas project has been filled with gasification slag of 3,000,000 tons, thermoelectric slag of 170,000 tons and fly ash of 210,000 tons, a large amount of solid waste has not been recycled. Based on the fact that solid wastes such as slag with good recycling properties, and combined with the problems of high organic concentration and difficult degradation of the coal-to-gas wastewater, in this paper, a new method of treating refractory organic wastewater from coal gasification with the compound of waste slag and fly ash is proposed. The effects of different factors on the removal of COD, total phenol and turbidity in wastewater were studied. Furthermore, based on the composite characteristics of adsorption and coagulation of this kind of solid waste, the economy of its combination with the existing coagulation process was explored.

2. Experimental

2.1. Experimental Wastewater Source and Quality

The experimental wastewater comes from biochemical treatment effluent of a coal chemical wastewater treatment system. During the experiment, the water quality fluctuated greatly due to the change of production load. Specific indicators are shown in Table 1.

Table 1. *Experimental wastewater quality.*

indicators	CODcr /(mg/L)	Total phenol /(mg/L)	Turbidity /(NTU)	pH
range	321~961	28.4~62.4	141~202	8.3~8.7

2.2. Pretreatment of Solid Waste

The solid wastes used in this experiment come from the mixture of gasification slag, thermoelectric slag and fly ash in a coal chemical landfill site. After a certain amount of solid waste mixture is obtained, it is soaked in pure water, washed, and then dried. When the mass is constant, it is taken out for mechanical grinding. After mechanical grinding for a certain period of time, the composite with particle size less than 0.1mm is screened by a screen (the composite is hereinafter referred to as Fx). The density is 1 g/cm^3 and the specific surface area is 15235 cm^2/g, the main components include silicon dioxide, calcium oxide, alumina, iron oxide, etc, the specific composition and content are shown in Table 2.

Table 2. *Fx composition and content.*

composition	content (%)	composition	content (%)
SiO_2	62.1	MgO	4.7
CaO	9.2	TiO_2	1.9
Al_2O_3	9.6	Na_2O	1.2
Fe_2O_3	6.7	P_2O_5	0.5
SO_3	3.7	K_2O	0.4

2.3. Experimental Instruments and Reagents

The instruments used in this experiment mainly include: magnetic stirrer (Zhong xing Weiye Century Instrument 79-1); COD digestion apparatus (HACH DRB200); Uv-visible spectrophotometer (HACH DR6000); Turbidimeter (HACH 2100N), etc, as well as beaker, measuring cylinder, pipette and other glass instruments.

The reagents used in this experiment mainly include COD prefabricated reagent, 30% industrial hydrochloric acid, KBR-KBRO3 standard titration solution, $Na_2S_2O_3$ standard titration solution, polyaluminum chloride (PAC) and polyacrylamide (PAM), etc.

2.4. Experimental Methods and Procedures

Static experiment was adopted in this experiment. See Figure 1 for the specific process and Figure 2 for the experimental scene. First, the experiment of Fx alone treatment of wastewater was carried out, take a certain volume of refractory organic wastewater into a beaker, add a certain quality of Fx, stir for a certain time, and then stand for precipitation. After the precipitation, the COD concentration, total phenol concentration and turbidity of the supernatant were measured, and the removal effect of Fx on pollutants was analyzed. Then, according to the characteristics of Fx, the combined experiment with coagulant was carried out, take a certain volume of refractory organic wastewater into the beaker, add a certain amount of Fx for a certain time after stirring, and then add a certain amount of coagulant for coagulation precipitation; After the precipitation is complete, the supernatant is taken to test its COD concentration, total phenol concentration and turbidity, and the combined effect of Fx and coagulant is analyzed.

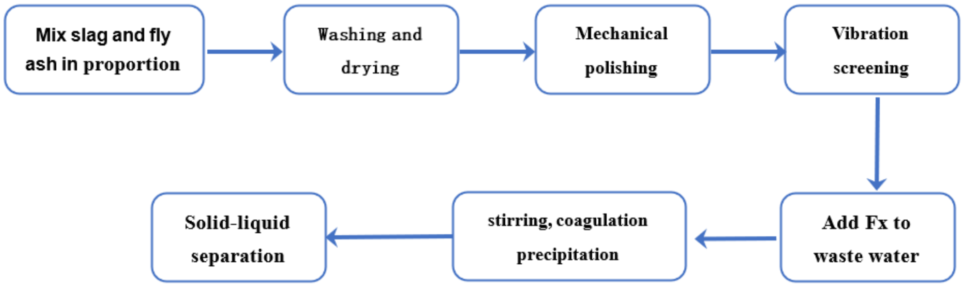


Figure 1. *Experimental flow chart.*

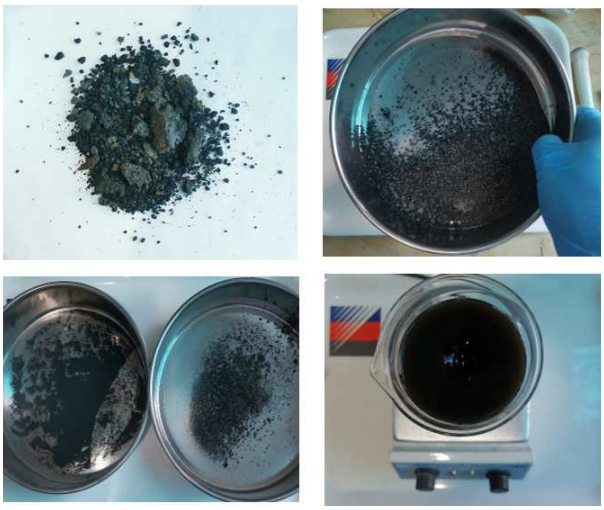


Figure 2. *Experimental scene.*

COD was tested by potassium dichromate method, the turbidity was determined by HACH-2100N turbidimeter. Total phenol concentration was measured by bromination volumetric method, its specific steps are: 1、Pipette 50 mL sample (or 50 mL diluted water sample) into 250 mL iodine flask, add 1+1 sulfuric acid 10 mL, shake the Iodine volumetric flask slowly; 2、add 25 mL KBr-$KBrO_3$ standard titration solution with water seal, place in dark place for 1h, then add 10 mL KI solution and seal with water, continue to hide from light for 10 min, titration was performed with $Na_2S_2O_3$ standard titration solution, 1 mL starch indicator was added near the end of titration, continue dripping until the blue is gone, record the volume of $Na_2S_2O_3$ standard titrated solution consumed, used 50 mL distilled water to replace the sample for blank test. The formula for calculating the total phenol concentration is:

$$\text{The total phenol concentration (mg/L)}=\frac{c(V_0-V_1)\times 15.68}{V}\times 10^3$$

In the type:

c——The concentration of $Na_2S_2O_3$ solution, mol/L;

V_0—— Blank test consumes the volume of $Na_2S_2O_3$ standard titrated solution, mL;

V_1—— The sample to be measured consumes the volume of $Na_2S_2O_3$ standard titrated solution, mL;

V——volume of the sample, mL;

15.68——1/6 (C_6H_5OH) Molar mass, g/mol.

3. Experimental Results and Analysis

3.1. Influence of Dosage

When the COD concentration of wastewater was 624 mg/L, the total phenol concentration was 60.5 mg/L, and the turbidity was 92.9 NTU, pour 500 mL of waste water into a beaker, add 5 g/100 mL, 10 g/100 mL, 15 g/100 mL, 20 g/100 mL and 25 g/100 mL Fx respectively, stirring for 30 min at a rotational speed of 500 r/min, then let it rest sufficiently, take the supernatant to test COD concentration, total phenol concentration and turbidity, the results were shown in Figure 3.

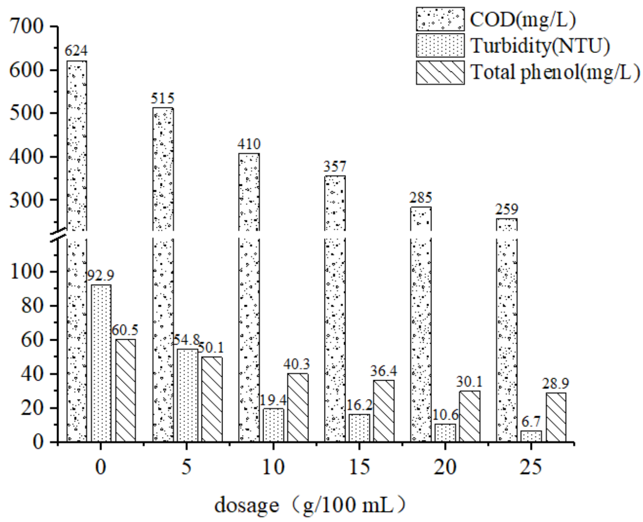


Figure 3. *Influence of dosage on pollutant removal characteristics.*

It can be seen from Figure 3, COD concentration, total phenol concentration and turbidity of supernatant decreased with the increase of Fx dosage, indicating that the greater the Fx dosage. The better the removal effect of wastewater pollutants, the removal rate of COD, total phenol and turbidity can reach 57.5%, 52.2% and 92.8% respectively. When the dosage of Fx is greater than 20 g/100 mL, the degree of improvement of removal effect of COD, total phenol and turbidity decreases slightly, mainly because Fx has strong adsorption and flocculation. When the dosage reaches 20 g/100 mL, the two effects reach a relative balance. Therefore, according to the feasibility of actual operation and the requirements of removal effect, 20 g/100 mL is a more appropriate dosage, at this time, the removal rate of COD is 54.3%, total phenol is 50.2%, turbidity is 88.6%.

3.2. Influence of Stirring Time

When the COD concentration of wastewater is 961 mg/L, the total phenol concentration is 62.4 mg/L, and the turbidity is 152 NTU, pour 500 mL of waste water into a beaker, add 20 g/100 mL Fx, at a stirring rate of 500 r/min. The mixture was stirred for 5 min, 10 min, 15 min, 20 min, 25 min and 30 min respectively, explore the influence of stirring time on the removal effect of COD, total phenol and turbidity, the results were shown in Figure 4.

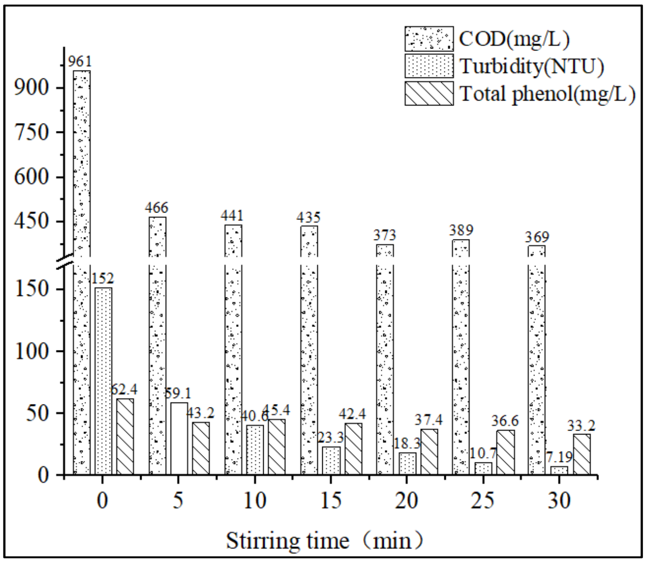


Figure 4. *Influence of stirring time on pollutant removal characteristics.*

It can be seen from Figure 4, after adding Fx, the removal effect of COD and total phenol concentration in the supernatant increased slightly with the increase of stirring time, while the removal effect of turbidity increased significantly with the increase of stirring time. The results showed that stirring time had little effect on the removal of COD and total phenol by Fx, but had great effect on the removal of turbidity. Within 5~30 min, the COD removal rate is in the range of 49.4%-61.6%, and the total phenol removal rate is in the range of 27.2%-46.8%. While the turbidity removal rate increased 34.2% from 61.1% when stirring for 5 min to 95.3% when stirring for 30 min.

Analysis of the reasons shows that Fx is mainly used for adsorption of dissolved organic matter, At the beginning, Fx has strong adsorption capacity and great adsorption power, and reaches a relative adsorption equilibrium state in a relatively short time, when Fx mainly flocculates and precipitates particles and colloids in wastewater, With the increase of stirring time, Fx gets more and more exposed to particulate matter, and the flocs formed are more and more dense, that is beneficial to the separation and removal of pollutants. Therefore, the turbidity removal rate increases the most with the increase of stirring time, and prolonged stirring time is more conducive to the removal of wastewater turbidity.

3.3. Influence of pH

When the COD concentration of wastewater is 853 mg/L, the total phenol concentration is 61.3 mg/L, the turbidity is 146 NTU, the pH is about 8.5, and the Fx dosage is 20 g/100 mL, the stirring rate is 500 r/min, and the stirring time is 5 min, the pH of raw water was adjusted to 8.5, 7.0, 5.5, 4.0 and 2.5 by adding 30% industrial hydrochloric acid to the wastewater, the effect of pH on the removal of COD, total phenol and turbidity by Fx was explored. The results were shown in Figure 5.

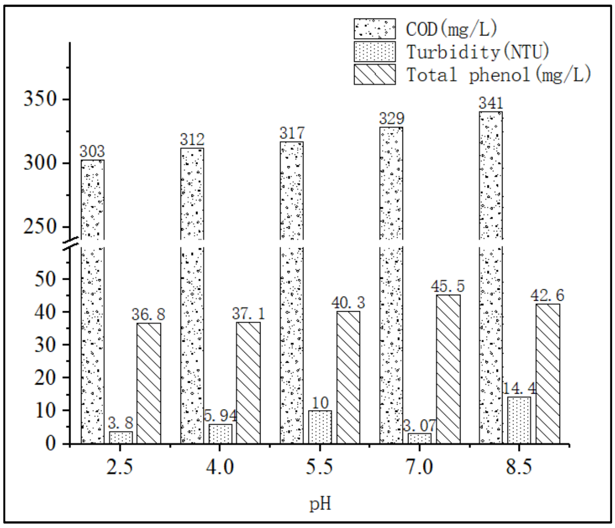
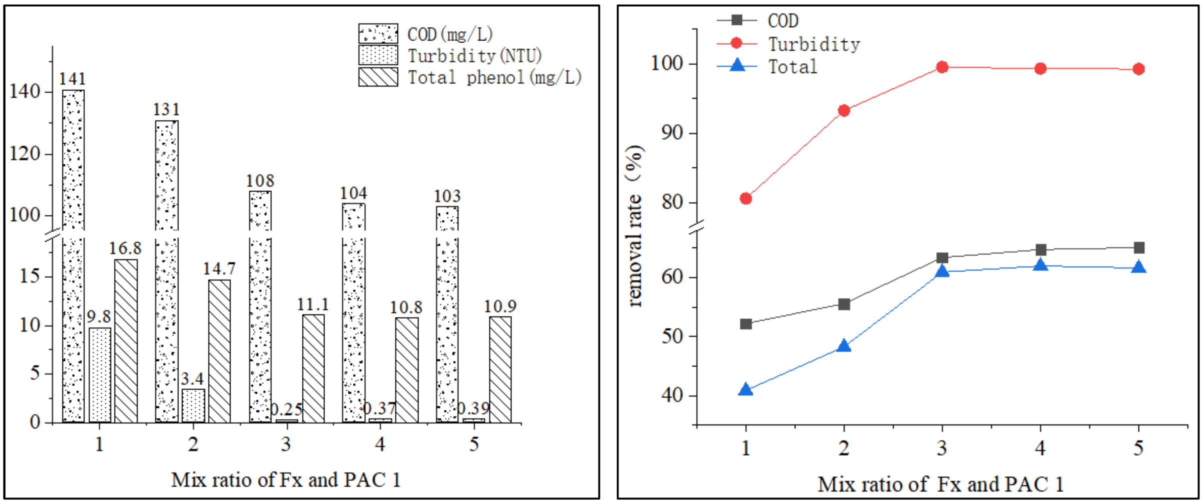


Figure 5. *Influence of pH on pollutant removal characteristics.*

It can be seen from Figure 5, When the pH of waste water is adjusted by adding industrial hydrochloric acid, COD in supernatant decreased from 341 mg/L at pH 8.5 to 303 mg/L at pH 2.5, its removal rate increased from 60% to 64.5%. the total phenol concentration decreased from 42.6 mg/L at pH 8.5 to 36.8 mg/L at pH 2.5, its removal rate increased from 30.5% to 39.9%. The removal rate of COD and total phenol only increased by 4.5% and 9.4% respectively. The turbidity of supernatant showed two lows with the change of pH, when pH is 7, turbidity removal rate is 97.9%, and when pH is 2.5, turbidity removal rate is 97.4%. According to the analysis, when the pH is 7, the flocculation of aluminum ions and iron ions dissolved by Fx can be more fully played, when the pH is 2.5, Fx releases more aluminum and iron ions, the turbidity removal effect was enhanced by increasing the concentration of coagulation components. Therefore, it can be seen that the improvement effect of changing the pH of wastewater on Fx removal of COD and total phenol is not very obvious, while there are various effects on turbidity removal, considering the operating conditions and costs, the pH of wastewater is more appropriate between 6.5 and 7.5.

3.4. Synergy Between Fx and PAC

The traditional coagulation process with PAC as coagulant is a common technology in wastewater treatment process, which is mostly used after biochemical treatment. Its main principle is to destroy the stability of colloid by adding chemical agents to wastewater, so that colloid and fine suspended particles gather into flocculation, separation. In addition to the addition of coagulants, the typical high-efficiency coagulation and precipitation process also quantitatively adds granular media (such as sludge in sedimentation tank or tiny sand) that can improve the coagulation effect to increase the concentration of suspended solids in raw water, increase the reaction rate and shorten the reaction time. At the same time, sand particles are used as the core of flocculation to form a more dense flocculant with better sedimentation performance, which can improve the removal effect of turbidity in wastewater [17].



(a) (b)

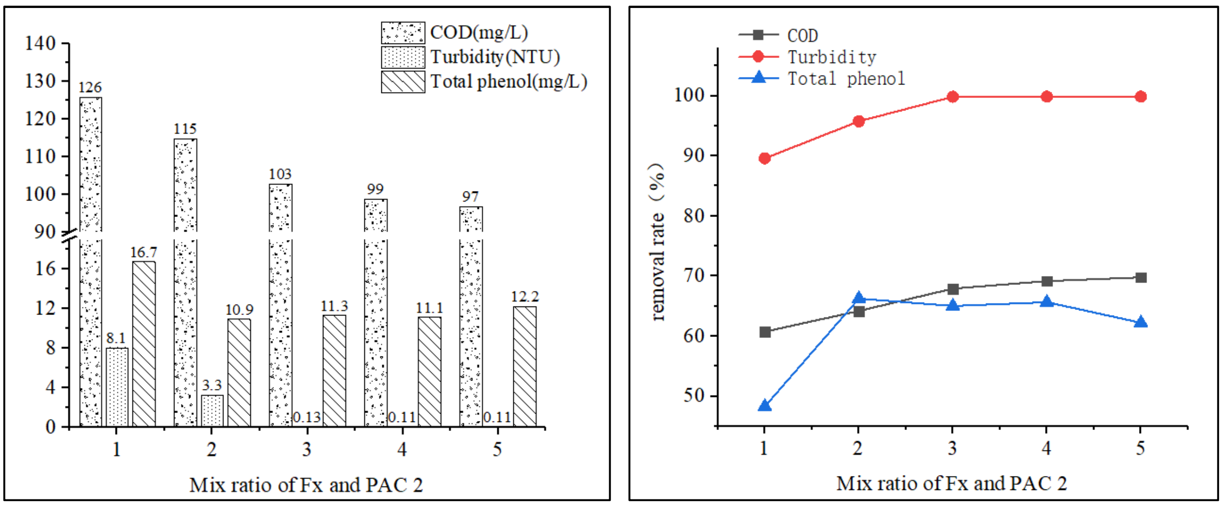
(Note: The COD of waste water is 295 mg/L, the total phenol concentration is 28.4 mg/L, and the turbidity is 50.5 NTU.)

Figure 6. *(a). Concentrations of pollutants in the supernatant at different mix ratios. (b) Removal efficiency of each pollutant in supernatant at different mix ratio.*

On the basis of traditional coagulation process, the combined experiment of Fx and PAC was carried out in this experiment. The changes of COD concentration, total phenol concentration and turbidity in wastewater under different combinations of Fx dosage and PAC dosage were investigated. Take a certain amount of waste water into the beaker, first add Fx with a certain concentration, and stir for 5 minutes, then continue to add PAC with a certain concentration, and stir for 5 min. After standing the precipitation, take the supernatant to test COD concentration, total phenol concentration and turbidity., and calculate the removal efficiency of each pollutant respectively, the results are shown in Figure 6-a and 6-b and Figure 7-a and 7-b, the corresponding combined ratio of Fx dosage and PAC is shown in Table 3 and Table 4.

Table 3. *Mix ratio of Fx and PAC 1.*

The number	Mix ratio 1
1	0.5 ppm PAM+200 ppm PAC
2	5 g/100 mL Fx+200 ppm PAC
3	10 g/100 mL Fx+200 ppm PAC
4	15 g/100 mL Fx+200 ppm PAC
5	20 g/100 mL Fx+200 ppm PAC



(a) (b)

(Note: The COD of waste water is 321 mg/L, the total phenol concentration is 32.3 mg/L, and the turbidity is 77.8 NTU.)

Figure 7. *(a). Concentrations of pollutants in the supernatant at different mix ratios.(b). Removal efficiency of each pollutant in supernatant at different mix ratio.*

Table 4. *Mix ratio of Fx and PAC 2.*

The number	Mix ratio 2
1	10 g/100 mL Fx＋0 ppm PAC
2	10 g/100 mL Fx＋25 ppm PAC
3	10 g/100 mL Fx＋50 ppm PAC
4	10 g/100 mL Fx＋100 ppm PAC
5	10 g/100 mL Fx＋200 ppm PAC

According to Table 3 and Figure 6-a and 6-b, when PAC dosage is fixed at 200 ppm, the COD concentration, total phenol concentration and turbidity of supernatant decreased gradually, and the corresponding removal efficiency improved gradually with the increase of Fx concentration. The effluent effect is better than that of traditional PAC+PAM. When the Fx dosage is greater than 10 g/100 mL, COD, total phenol and turbidity in the supernatant are basically unchanged, and the corresponding removal rate tends to be gentle. It shows that 10 g/100 mL Fx dosage is suitable, while the removal rate of COD, total phenol and turbidity reached 63.4%, 60.9% and 99.5% respectively.

It can be seen from Table 4 and Figure 7-a and 7-b, when Fx dosage is 10 g/100 mL, PAC dosage increases from 0 to 50 ppm, the COD concentration, total phenol concentration and turbidity of supernatant decreased gradually, while the removal rate of each pollutant increased gradually. After continue to increase PAC dosage, COD, total phenol and turbidity of supernatant did not improve significantly, and the removal rate of total phenol showed a downward trend. Therefore, 10 g/100 mL of Fx dosage and 50 ppm of PAC dosage is the best combination, the removal rate of COD, total phenol and turbidity reached 67.9%, 65% and 99.8% respectively, compared with the effect of the combination of PAC and PAM. The removal rates of COD, total phenol and turbidity increased by 15.7%, 24.2% and 19.3% respectively.

4. Economic Analysis

Based on the analysis of a coal-to-gas industrial wastewater project, its wastewater design flow is 1200 m^3/h, PAC and PAM whose dosages are 200 ppm and 0.5 ppm are currently selected as additives for the coagulation process in its wastewater treatment system. According to the combined experiment of Fx and PAC. the combination of Fx (10 g/100 mL) and PAC (50 ppm) can not only improve the removal efficiency of COD, total phenol and turbidity, but also save at least 75% of PAC dosage compared with the existing coagulant dosage combination. The market price of PAC with 28% active component is about 3100 yuan per ton, and the operating time of the wastewater treatment system is calculated by 300 days/year, then the PAC consumption can be reduced by about 1300 tons per year, that is, the direct cost of PAC agent can be saved at least more than 4 million yuan. Reducing the dosage of reagents can also reduce the sedimentation sludge, which can effectively reduce the overall operation cost of wastewater, and has great economic value and application prospect.

5. Estimate of Carbon Reduction Effect

In the process of the original method of industrial wastewater treatment of the coal to gas project, the average monthly dosage of PAC was about 173t, the average monthly sludge production was about 500t. When Fx was used to replace PAC, the generated sludge could return to the biochemical system to play the role of biological carrier, which could greatly reduce the disposal amount of the original biochemical sludge. Therefore, when the amount of PAC was reduced by 1300 t/a, the corresponding sludge could be reduced to produce 3757 t/a (90% moisture content).

At present, the conventional disposal method of sludge in this project is "drying + sending to the boiler in the plant for incineration". Carbon emission mainly comes from heat energy consumed by drying and CO_2, CH_4 and NO_2 produced by incineration. According to the calculation method and discharge factor provided by IPCC, the carbon emission of dry incineration after deep dehydration of sludge to 60% moisture content was calculated, total carbon emission of per ton dry sludge is 3575.7 kg (in CO_2) [18]. When 3757 t of sludge with 90% moisture content can be reduced annually, the project can reduce about 1343 t of CO_2 emissions annually.

6. Conclusion

1) Fx has good removal effect of COD, turbidity and the refractory organic compounds- total phenols of organic wastewater. When the pollutants in organic wastewater are treated by Fx alone, the best removal rate of COD, total phenol and turbidity reaches 60%, 50.2% and 97% respectively. The COD concentration is reduced from 853 mg/L to 303 mg/L, and the removal amount reaches 550 mg/L. The total phenol concentration is reduced from 60.5 mg/L to 30.6 mg/L, and the removal amount reaches 29.9 mg/L, the turbidity is reduced from 146 NTU to 3 NTU, and the removal is 143 NTU.
2) According to the single factor experiment of Fx, the additional concentration of Fx has a great influence on the overall removal of pollutants, while the stirring time has little influence on the removal of COD and total phenol, which has a great influence on the removal of turbidity. The pH has a small influence on the overall removal of pollutants. Comprehensively considering the operation management and removal effect requirements of wastewater treatment process, the additional concentration of Fx should be 20 g/100 mL, the stirring time should be 30 min, and the pH should be about 6.5~7.5.
3) Fx is used in combination with PAC, on the one hand, it accelerates the precipitation rate of the flocculant and reduces the precipitation time. On the other hand, it also improves the removal effect of the pollutants in the refractory organic wastewater, compared with the original coagulation process. The COD removal rate,

total phenol removal rate and turbidity removal rate can be increased by 15.7%, 24.2% and 19.3% respectively.

4) Fx has great economic value, combining Fx and PAC for the treatment of refractory organic wastewater. Consequently, this research makes it possible to reduce the cost of solid waste disposal, the dosage of PAC agent by 1300 tons every year, and to save at least 4 million yuan PAC agent direct cost as well.
5) Using Fx to treat refractory organic wastewater greatly reduces the storage of solid waste and improves the efficiency of resource reuse. Moreover, 3757 t of sludge with 90% moisture content can be reduced annually, and about 1343 t of CO_2 emissions can be reduced at the same time, which has a good carbon emission reduction effect, contributing to the realization of the low-carbon green cycle development concept of “treating waste with waste”.

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