

Influence of Humic Substance in Solids on the Measurement of Oxygen-Consuming Organics of the Yellow River

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ABSTRACT. The Yellow River has a high content of solids. The influence of solids on the measurement of oxygen-consuming organics (OCOs) such as chemical oxygen demand (COD_{Mn}) has aroused attentions in recent years. However, no research about effects of humic substance in solids on the determination of COD_{Mn} has been reported. Experiments have been carried out to study the influence of humic substance in solids (Loess soil) on COD_{Mn} in this research. Several significant results have arisen from the study. (1) The total COD_{Mn} of water sample including liquid and solid phases increased significantly with the increase of solid content; the COD_{Mn} of liquid phase of water sample under pre-treatment with acid (LPUPA) also increased significantly with the increase of solid content while that without pre-treatment did not increased significantly. (2) With the increasing of solid content, the total BOD_5 of water sample increased much slower than the total COD_{Mn} and the COD_{Mn} of LPUPA. (3) The humic substances in solids contributed greatly to the total COD_{Mn} of water sample. When the solid contents were 7.5 and 15.0 g/L, the humic substances in solids accounted for 15.9% and 17.1% of the total COD_{Mn} of water sample, respectively; fulvic acid-one of the main compositions of humic substance-contributed to 23.6% and 46.3% of the COD_{Mn} of LPUPA, respectively. (4) The humic substances can hardly be biologically oxidized under natural conditions, the COD_{Mn} of LPUPA and the total COD_{Mn} of water sample exaggerates the oxygen-consuming organic contamination of the Yellow River. Therefore, COD_{Mn} was not suitable for being regarded as a parameter reflecting the pollution degree of OCOs in river water with a high solid content.

Keywords: COD_{Mn} , BOD_5 , humic substances, solids, Yellow River

1. Introduction

The Yellow River is the second longest river in China. It provides important water resources to the northern and northwestern regions of China. However, the water quantity is very scarce. In addition, deteriorating water quality of the Yellow River has been intensifying water shortage in the last decade (Zhao et al., 1998; Zhu et al., 1999; Xia et al., 2002); the functional water volume has thus been decreasing.

Oxygen-consuming organics (OCOs) is one of the main pollutants in the river. Chemical oxygen demand (COD_{Mn}) and biological oxygen demand (BOD_5) have been used as the water quality parameters to reflect the pollution degree of OCOs. COD_{Mn} refers to the amount of oxygen consumed when the organic matter in a given volume of water is chemically oxidized to CO_2 and H_2O by permanganate, a strong chemical oxidant. BOD_5 refers to the amount of oxygen consumed when the organic matter in a given volume of water is biodegraded for five days. COD_{Mn} is determined through oxidizing water sample with chemical oxidant while BOD_5 with microorganism. Therefore, BOD_5 directly reflects the pollution degree of OCOs while COD_{Mn} indirectly reflects the pollution degree. They are coincident under normal conditions.

However, the measurement values of COD_{Mn} did not coincide with those of BOD_5 for the Yellow River. As the content of suspended solids is very high in the river, with an average suspended solids content of 28 g/L (Zhao, 1998), the influence of solids on the measurement of OCOs, especially on COD_{Mn} value, has aroused attention from the country in the last decade (Wang, 1995). For example, Li Qingfu and Hu Guohua has studied the relationship between COD_{Mn} and solids (Li and Hu, 1997; Hu, 2000), and the research results indicated that total COD_{Mn} value of water sample including liquid and solid phases increased greatly with the solid content. Recently, Chen et al. have studied the influence of solid on COD values (Chen et al., 2003, 2004); based on the previous measurement data of COD_{Mn} , BOD_5 and solid content of the river, they conducted correlation analysis and suggested that humic substance in solid was the key factor affecting the determination of COD_{Mn} value.

In addition, during the past decades, many scientists around the world have carried out researches to study the influences of OCOs on dissolved oxygen (DO) and relationships between DO and BOD as well as between BOD and COD in waste and natural waters (Adrian and Sanders, 1998; Leu et al., 1998; Baker et al., 1999). Some researchers have studied methods for determination of COD in natural waters (Nabivanets et al., 2001; Udovichenko et al., 2001).

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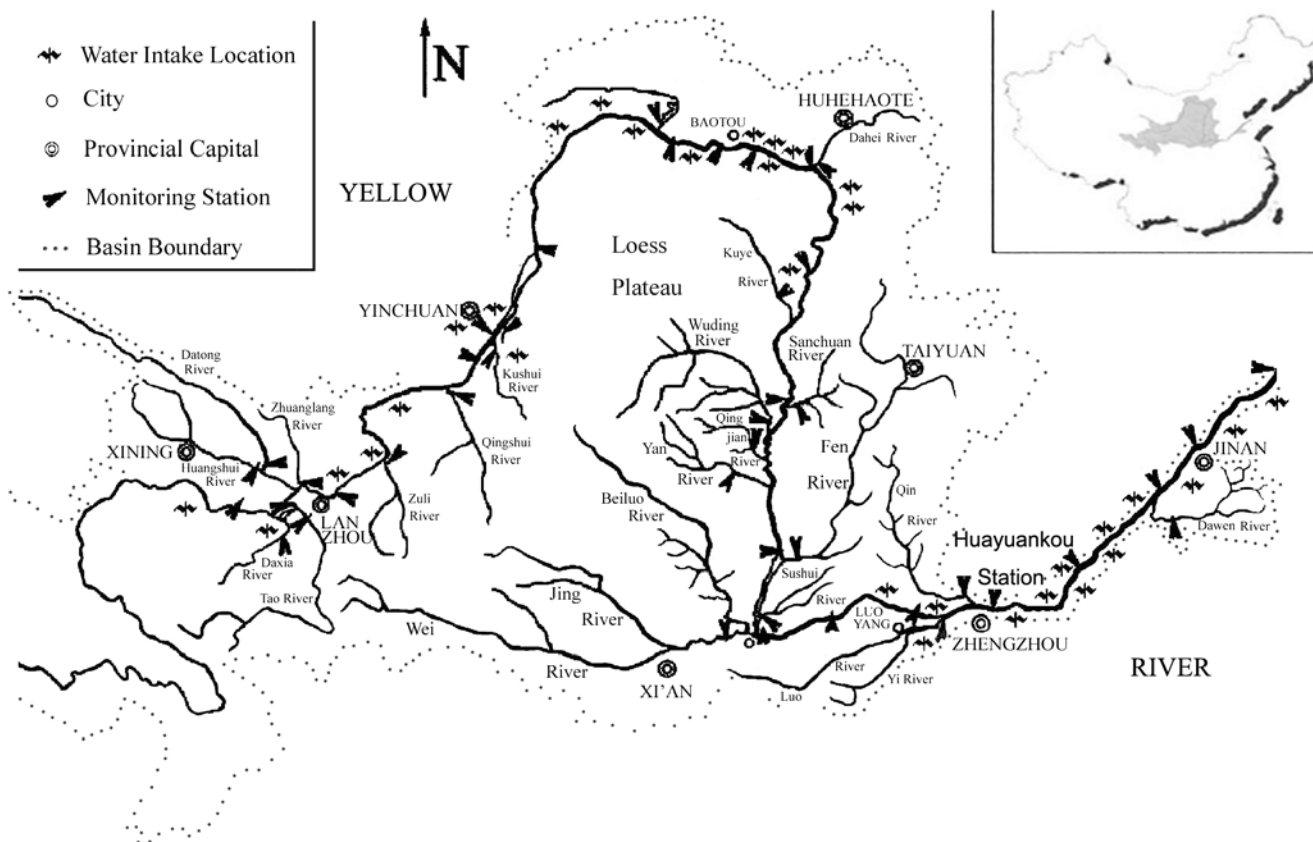


Figure 1. The Yellow River basin and the sampling locations.

However, no previous experimental research has been carried out to study the effects of humic substances on the measurement of OCOs of a river with high content of suspended solids. In addition, we don't know which part of humic substances in solid exerts influence on COD_{Mn} and BOD_5 values of a river, and in-depth analyses on strength of the influence are desired.

Therefore, this study is an extension of the previous effort, emphasizing on studying the influence of humic substances on the measurement of OCOs of the Yellow River through simulation experiments. The effects of each composition in humic substances will be analyzed; the contribution of the main influencing composition to COD_{Mn} value in river water will be addressed. At the same time, biodegradation of humic substances in the solid will be studied to find out whether they fall within oxygen-consuming organics.

2. Material and method

2.1. Sample Collection

In this study, the sampling site of river water was located at the Huayuankou Hydrological Station which is in the middle reach of the Yellow River (Figure 1). Water samples were collected at a depth of 0.2 m in the central stream point on

November 19, 2002 and February 24, 2003. All samples were maintained at 4°C in a cooler, and then transported to the laboratory. After being centrifuged at 4000 rpm for fifteen minutes (which was sufficient to remove all solids), the supernatant of the water sample was decanted for experiment work. Since solids in river water mainly come from the Loess Plateau which is located in the middle reach of the Yellow River, the solid (soil) sample site was located at Suide and Ansai of the plateau. Three kinds of soils were collected from Ansai, which included the soils without cultivation for 20, 8 and 2 years, respectively. Soil samples were collected at a depth of 0-20 cm on August 10, 2002. All the soil samples were air-dried and grounded, then sieved through 0.154 mm sieve for experimental analyses. The soil samples collected from Suide were used to study the effects of solids on the determination of COD_{Mn} and BOD_5 in water system; the soil samples collected from Suide and Ansai were used to study the biodegradation of humic substances in solids under natural conditions.

2.2. Experimental Design

(1) Effect of Solid Content on the Determination of COD_{Mn} and BOD_5

Since the solid content of river water at the Huayuankou

Station ranged from 0.4 to 15.8 g/L, influences of 0, 7.5, 10 and 15 g/L solids on the measurement of COD_{Mn} and BOD_5 of river water samples were studied. For the influence of solids on BOD_5 , a known amount of soil sample was added to a series of 500 mL supernatant of river water sample, and stirred with magnetic agitator for an hour. Then BOD_5 value of sample including liquid and solid phases was determined; after being centrifuged at 4000 rpm for fifteen minutes, the supernatant of the water sample was decanted for the determination of BOD_5 value.

In the regular measurement procedure of the Yellow River, acid was added to river water sample immediately after the collection; this was intended to prevent the organics from being biodegraded. Samples were then transported to the laboratory to determine COD_{Mn} values of the supernatant and the sample including liquid and solid phases. Therefore, the COD_{Mn} values of samples under pre-treatment with acid were also determined in this study to follow the routine measurement procedure of the river. For the influence of solids on COD_{Mn} , a known amount of soil sample was added to a series of 100 mL supernatant of river water sample, and sulfuric acid was added to the mixture of water and soil samples to make the pH value equal to 2.0. After that, the flasks were stirred with magnetic agitator for an hour. Then total COD_{Mn} value of sample including liquid and solid phases was determined; after being centrifuged at 4000 rpm for fifteen minutes, the supernatant of the water sample was decanted for the determination of COD_{Mn} value. In addition, COD_{Mn} values of samples without pre-treatment with acid were also determined in this research to study the effect of pre-treatment with acid on the determination of COD_{Mn} . Each experimental set was conducted in duplicate with a set of controls. The control experiments were carried out with water samples without addition of soil samples.

(2) Contribution of Humic Substances in Solids to COD_{Mn} Value

Humic substances extracted from 7.5 and 15 g soil samples were added to 1000 mL supernatant of river water sample, respectively, and stirred with magnetic agitator for an hour. COD_{Mn} values of the samples were then measured and COD_{Mn} value produced from humic substances could then be determined. Each experimental set was conducted in duplicate with a set of controls. The control experiments were carried out with water samples without addition of humic substances.

(3) Contribution of Fulvic Acid in Humic Substances of Solids to COD_{Mn} Value

Fulvic acid extracted from 7.5 and 15 g soil samples were added to 1000 mL supernatant of river water samples, respectively, and stirred with magnetic agitator for an hour. COD_{Mn} values of the samples were then measured and COD_{Mn} produced from fulvic acid could then be determined. Each experimental set was conducted in duplicate with a set of controls. The control experiments were carried out with water

samples without addition of fulvic acid.

(4) Biodegradation of Humic Substances

Natural river water and soil samples were used as media for biodegradation experiments. A total amount of 7.5 and 15 g soil samples were added to a series of glass flasks that contained 1000 mL supernatant of river water samples, respectively. Then the flasks were covered by eight layers of gauze to prevent outside bacteria from entering them. They were cultivated in the dark for five days at 25°C with a HZQ-F incubator under constant shaking (150 strokes/minute) to promote the growth of indigenous microorganisms. Humic substances in samples including liquid and solid phases were measured before and after cultivation, respectively. Then the biodegradation efficiency of humic substances within five days was determined. Each experimental set was conducted in duplicate with a set of controls. The control experiments were carried out with the sterilized water and soil samples, and microbial activities were inhibited through mercuric chloride.

2.3. Chemical Analysis

The humic substances in solids were extracted by sodium pyrophosphate—sodium hydroxide (Wen et al., 1984). The humic substances in river water were extracted by XAD-8 resin and then eluted with sodium hydroxide solution (Tao et al., 1988). Fulvic acid was obtained after the acidification, filtration and purification of humic substances (Nanjing Institute of Soil Science, 1978). The obtained humic substances and fulvic acid were determined through Phoenix 8000 TOC instrument. The liquid phase of water samples were obtained after centrifugation with LD5-2A centrifugal machine. COD_{Mn} values of samples were measured by potassium permanganate with acidity (Xi et al., 2004). BOD_5 of samples were measured at 20°C with an AL320 BOD_5 machine made in WTW Company of Germany.

Samples were collected from the river for two times, as the results of these two samples were similar, only one set of data were shown in this paper. Each experimental set was conducted in duplicate; the relative standard deviation was less than 5%.

3. Results and Discussions

3.1. Water Sample Chemistry and Humic Substance Analysis

The ion concentrations in water samples were determined with ion chromatography. As shown in Table 1, the total ion concentration in the water sample was 532.4 mg/L. The pH value was 8.08, indicating that the water was slightly alkaline. The contents of fulvic acid and humic substances in soil sample were 0.28% and 0.76%, respectively. Therefore, the content of fulvic acid in humic substances was 36.8%. The chemical and physical characteristic of the solids in the river was shown in the previous study (Xia et al., 2004).

Table 1. Chemical Composition of the Water Sample Collected on November 19, 2002 (mg/L), except for the pH

pH	DO	COD _{Mn}	BOD ₅	NH ₄ ⁺ -N	Ca ²⁺	Mg ²⁺	K ⁺	Cl ⁻	SO ₄ ²⁺	HCO ₃ ⁻
8.08	10.0	8.5	4.0	2.34	66.7	18.2	71.4	82.8	106.5	184.5

3.2. Influence of Solids on COD_{Mn}

(1) Water Sample without Pre-treatment with Acid

As shown in Figure 2, the total COD_{Mn} of water sample including liquid and solid phases increased with the content of solids. When the solid contents were 0, 7.5, 10, and 15 g/L, COD_{Mn} values were 8.5, 41.2, 60.8, and 85.6 mg/L, respectively.

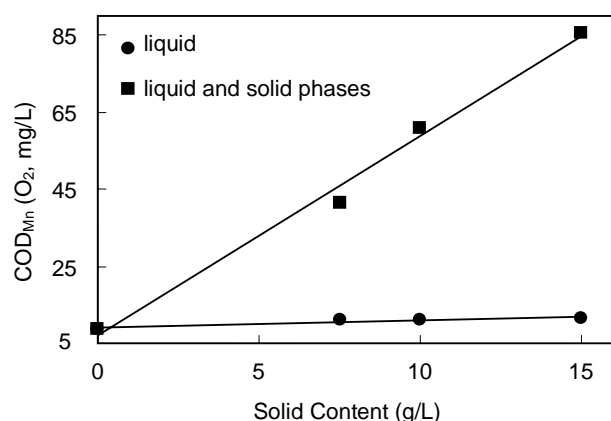


Figure 2. Effect of solids on COD_{Mn} value in water system (without pre-treatment with acid).

COD_{Mn} value of supernatant of water sample increased slightly with the content of solids. When the solid content increased from 0 to 7.5 and to 15 g/L, COD_{Mn} value of the supernatant only increased from 8.5 to 11.0 and to 11.3 mg/L, respectively. This experimental result is consistent with those obtained by Li and Hu (1997).

(2) Water Sample under Pre-treatment with Acid

As shown in Figure 3, with the increasing of solid content, the increasing rate of COD_{Mn} value of liquid phase of water sample under pre-treatment with acid (LPUPA) was much higher than that without pre-treatment with acid (LPWPA). When the increment of solid content was 1 g/L, the increments of COD_{Mn} values of LPUPA and LPWPA were 1.0 and 0.2 mg/L, respectively.

(3) Influence of Solids on BOD₅

As shown in Figure 4, both total BOD₅ of water sample including liquid and solid phases and BOD₅ of liquid phase of

water sample increased with the solid content; the increasing rate of the former was much higher than that of the latter. As shown in Figure 5, however, with the increasing of solid content, the increasing rate of total BOD₅ of water sample was much lower than that of the total COD_{Mn} of water sample and COD_{Mn} of LPUPA. The increment of solid content of 1 g/L could lead to the increment of total COD_{Mn} of water sample of 5.14 mg/L and COD_{Mn} of LPUPA of 0.97 mg/L; while could lead to the increment of total BOD₅ of only 0.53 mg/L.

As above mentioned, COD_{Mn} value of water sample including liquid and solid phases increased greatly with the increasing of solid content. This was due to the fact that the organics and other reducing substances existing in solids could be oxidized by potassium permanganate. The reason for the increasing of COD_{Mn} value of LPUPA with solid content was that some substances in solids could be dissolved in acid and oxidized by potassium permanganate. Since BOD₅ value reflects the oxygen quantity consumed by organics biodegradation, then according to the above experimental results, we can deduce that there are some substances in solids, which can be dissolved in acid and oxidized by chemical oxidant but can hardly be decomposed by microorganism under natural aquatic conditions.

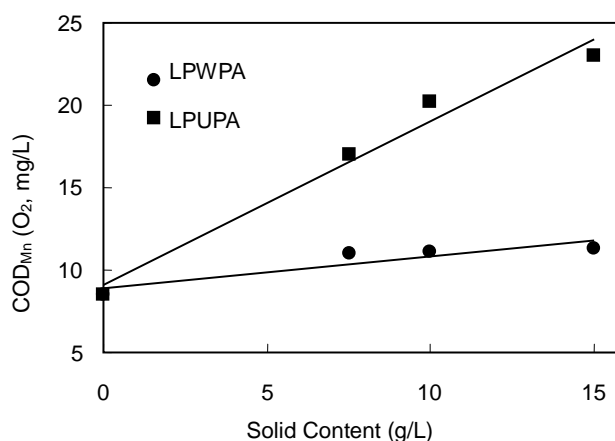


Figure 3. Comparison between effects of solids on COD_{Mn} values of LPUPA and LPWPA.

Humic substances are a naturally occurring mixture of organic compounds, which is slightly dissolved in water. According to its dissolution in acid and alkali, it can be divided into humic acid, fulvic acid and humus. The fulvic acid can be dissolved in acid and alkali; humic acid can only be

dissolved in alkali and humus can not be dissolved in acid or alkali. In addition, some research results indicated that humic substances is one kind of stable organics; it can hardly be degraded by microbes in natural system (Douglas et al., 1995; Sudha et al., 1995; Xiong et al., 1998; Huck, 1999). Therefore, we conjectured that the substances existing in solids, which can be oxidized by potassium permanganate and partly dissolved in acid but can hardly be biodegraded, is humic substances.

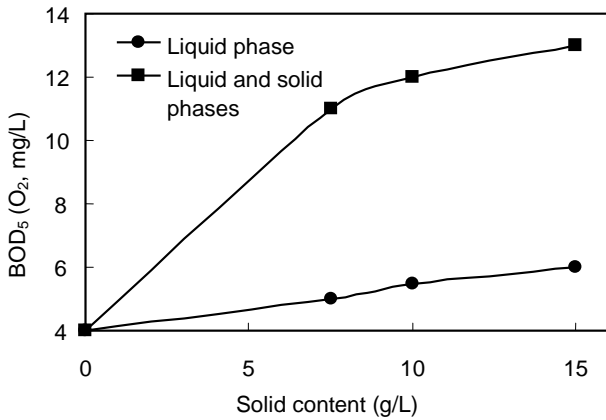


Figure 4. Relationship between solid content and BOD₅ values.

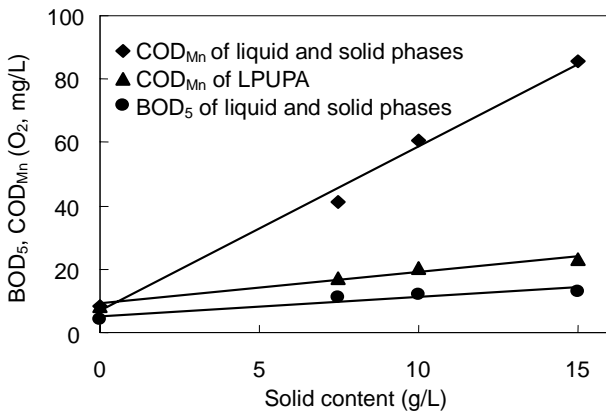


Figure 5. Comparison among effects of solids on BOD₅ and COD_{Mn} values.

(4) COD_{Mn} of Humic Substances in Solids

When the solid content was 7.5 g/L, COD_{Mn} value of humic substances in solids was 6.8 mg/L. It contributed to 15.9% of COD_{Mn} value (42.9 mg/L) of water sample including liquid and solid phases. When the solid content was 15.0 g/L, COD_{Mn} value of humic substances in solids was 14.6 mg/L. It contributed to 17.1% of COD_{Mn} value (85.6 mg/L) of water sample including liquid and solid phases.

As shown in Figure 3, COD_{Mn} value of LPUPA increased much more greatly than that of LPWPA with solid content. The contents of fulvic acid and humic substances in solids were 0.28% and 0.76%, respectively. Since fulvic acid is an organic component that can be dissolved in acid, we deduced that the notable increasing of COD_{Mn} value of LPUPA with solid content was caused by fulvic acid in solids. According to the experimental results, when the solid contents were 7.5 and 15.0 g/L, the COD_{Mn} values caused by fulvic acid in solids and the supernatant of river water sample were 12.5 and 19.2 mg/L, respectively; the COD_{Mn} values of LPUPA were 17.0 and 23.1 mg/L, respectively. The COD_{Mn} value of the supernatant of river water sample was 8.5 mg/L. Therefore, the COD_{Mn} values of fulvic acid in solids contributed to 23.6% and 46.3% of those of LPUPA, respectively.

As shown above, the COD_{Mn} values of fulvic acid in solids and the supernatant of river water were slightly less than those of LPUPA. This implied that there was other substances existed in solids which could be dissolved in acid and oxidized by chemical oxidant. The content of total organics in soil was 1.12%. As the content of humic substances in soil was 0.76%, the content of organics other than humic substances was 0.36%, which contributed to 32.1% of the total organics in soil. This kind of organics other than humic substance could also be oxidized by chemical oxidant, which led to the above phenomena that the COD_{Mn} values of fulvic acid in solids and the supernatant of river water were slightly less than those of LPUPA.

(5) Biodegradation of Humic Substances in Soil

A total amount of 7.5 and 15 g soil samples were added to 1000 mL supernatant of river water samples, respectively, and cultivated for five days at 25°C. The experimental results are shown in Figure 6. The sequence of humic substance content in soils was: soil without cultivation for 20 years > soil without cultivation for 8 years > soil without cultivation for 2 years. When the solid content was 7.5 g/L, the biodegradation rate of humic substance in solids ranged from 1.64% to 6.13%; when the solid content was 15.0 g/L, it ranged from 1.98% to 5.39%. Therefore, humic substances in the soil samples could hardly be biodegraded in natural conditions; that is to say, they could hardly consume oxygen in river water.

The humic substances with the content of 0.76% in solids (Loess soil) contributed greatly to the total COD_{Mn} of water sample. When the solid contents were 7.5 and 15.0 g/L, the humic substances in solids accounted for 15.9% and 17.1% of the total COD_{Mn} of water sample, respectively. In addition, as one of the main components of humic substances, fulvic acid can be dissolved in acid. Since acid is added to river water samples to prevent organic pollutants from being biologically oxidized before the determination of COD_{Mn}, fulvic acid existing in solids will be dissolved in water phase which is used to measure the COD_{Mn} of river water sample. COD_{Mn} value of LPUPA increased greatly with solid content. When the solid contents were 7.5 and 15.0 g/L, the COD_{Mn} values of fulvic acid in solids contributed to 23.6% and 46.3% of those

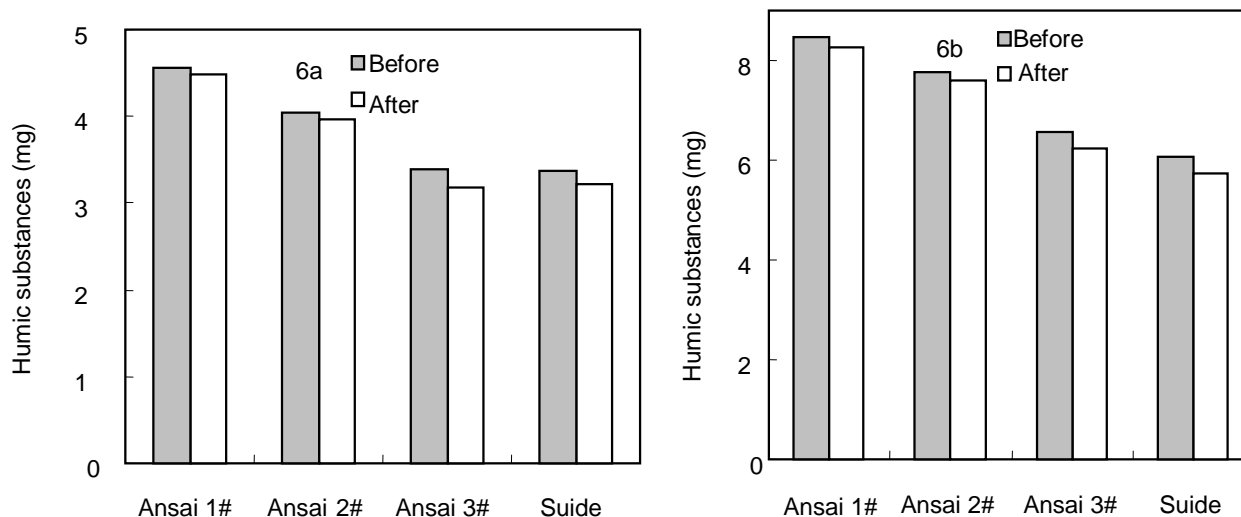


Figure 6. Biodegradation efficiencies of humic substances in soils collected from different places (6a: solid content was 5.0 g/L in water system; 6b: solid content was 10.0 g/L in water system. Ansai 1#, 2# and 3# refer to soil without cultivation for 20, 8 and 2 years, respectively).

of LPUPA, respectively. Based on the above-mentioned, the humic substances existing in solids can hardly be biologically oxidized in natural conditions. COD_{Mn} values of liquid phase and the whole water samples including liquid and solid phases influenced by the solids will exaggerate the OCOs pollution degree of the Yellow River; it is not suitable for being regarded as a water quality parameter reflecting the pollution degree of OCOs of the river.

4. Conclusions

Experiments have been carried out to study the influences of humic substances in solids (Loess soil) on the measurement of oxygen-consuming organics of the Yellow River in this research. The following results have arisen from the study:

(1) The total COD_{Mn} of water sample including liquid and solid phases increased significantly with the solid content; the COD_{Mn} of LPUPA also increased significantly with the increase of solid content while that of LPWPA did not increase significantly.

(2) With the increase of solid content, the increasing rate of total BOD_5 of water sample was much lower than those of COD_{Mn} of LPUPA and total COD_{Mn} of water sample.

(3) The humic substances with the content of 0.76% in solids (Loess soil) contributed greatly to the total COD_{Mn} of water sample. When the solid contents were 7.5 and 15.0 g/L, the humic substances in solids accounted for 15.9% and 17.1% of the total COD_{Mn} of water sample, respectively; fulvic acid — one of the main components of humic substance — contributed to 23.6% and 46.3% of the COD_{Mn} of LPUPA, respectively.

Since the humic substances can hardly be biologically

oxidized under natural conditions, the COD_{Mn} of LPUPA and the total COD_{Mn} of water sample exaggerated the oxygen-consuming organic contamination of the Yellow River. Therefore, COD_{Mn} is not suitable for being regarded as a parameter reflecting the pollution degree of OCOs in river water with a high solid content.

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