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Integrated assessment for aerobic biodegradability of sulfide mineral flotation collectors

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ABSTRACT

The biodegradability of flotation collectors has the most important impact on mine environment. Understanding the flotation collectors' biodegradability is a crucial element in developing an informed strategy for analysing and managing the mine environment. Integrated assessment method including three biodegradability tests namely, BOD_5/COD_{Cr} ratio test, Static-flasks test and OECD 301B have been established to evaluate the aerobic biodegradability of the sulfide mineral flotation collectors. Consistent conclusions have been obtained through these three biodegradability tests: the aerobic biodegradation of the different sulfide mineral flotation collectors is different, namely, sodium diethyldithiocarbamate is a readily biodegradable collector, and ammonium butyl-dithiophosphate is partially biodegradable. However, n-butyl xanthate and ethylthionocarbamate are considered as poorly biodegradable. Besides, the order of aerobic biodegradability of these collectors is: sodium diethyldithiocarbamate > ammonium butyl-dithiophosphate > n-butyl xanthate > ethylthionocarbamate.

Keywords: Sulfide mineral flotation collectors; Aerobic biodegradability; BOD_5/COD_{Cr} ratio test; Static-flasks test; OECD 301B

1. Introduction

Biodegradability is one of the most important characteristics of an organic compound for predicting its fate and life in the environment and its application in biological wastewater treatment [1]. The biodegradability of chemicals is one of the most important aspects of their environmental behaviour, because a biodegradable substance is expected to cause less ecological problems in the long term than a persistent one [2].

Sulfide mineral flotation collectors are the group most widely used in flotation [3]. Normally, they are discharged directly into the environment without any treatment. It has been known that even smaller

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concentrations of these reagents in water streams are toxic to water life, besides their deleterious influence on end-stream processes during recycling [4]. Serious environmental problems associated with the flotation reagents in water from mineral processing plant have been well documented [5].

Many assessment methods for evaluating the biodegradability of an organic compound have been described, including Zahn–Wellens test, BOD_5/COD_{Cr} ratio test, static-flasks test, the well-known and much-used CO_2 evolution test [6,7], manometric respirometry test, etc. [9].

The chemical-oxygen demand with potassium dichromate method (COD_{Cr}) determines the organic content in terms of both biodegradable and non-biodegradable compounds, whereas five-day biochemical oxygen demand (BOD_5) test evaluates the biodegradable fraction of the wastewaters [10]. Therefore, BOD_5/COD_{Cr} ratio constitutes a good measure of the biodegradability of a wastewater [11,12].

Static-flasks test is commonly used for the evaluation of the biodegradation potential of non-volatile organic compounds.

The Organisation for Economic Cooperation and Development (OECD) has made leading international efforts to standardize biodegradation test methods. The well-established OECD carbon dioxide method based on Sturm's original test is usually used for assessing biodegradability of more or less soluble organic chemicals [13]. The method has been internationally validated by a number of laboratories and it has been adapted as a new work item for the OECD Test Guideline for chemicals testing [14].

However, studies on the biodegradability of flotation reagents have seldom been reported. The existing research only focuses on photodegradation techniques. However, these methods are obviously deficient, such as high energy consumption, high operating costs, and may cause secondary pollution. Aerobic biodegradability techniques have gained attention as they are found to be versatile, inexpensive, stable and environmentally benign techniques in wastewater treatment. Therefore, the evaluation of biodegradability of flotation collectors could be contributed to provide some guidance to develop low toxicity and environmental-friendly flotation collectors, which are expected to play an important role in mine environmental protection.

In the present investigation, the biodegradability of typical sulfide mineral flotation collectors, namely sodium diethyldithiocarbamate, ammonium butyldithiophosphate, n-butyl xanthate and ethylthionocarbamate, have been evaluated according to BOD_5/COD_{Cr}, static-flasks test and OECD 301B, while the biodegradabilities were compared by different biodegradability tests.

2. Materials and methods

2.1. Materials

The analytical-grade reagents were purchased from Tianjin Chemical Co., Ltd. (Tianjin, P.R. China). Sulfide mineral flotation collectors (Structures are indicated in Fig. 1) were obtained from Zhuzhou Mineral Processing Reagent Plant (Zhuzhou, P.R. China).

2.2. Biodegradability measurements

2.2.1. BOD_5/COD_{Cr} ratio test

COD_{Cr} and BOD_5 were measured according to standard methods [15,16]. And the concentrations of the sulfide mineral flotation collectors were $30\,mg\,L^{-1}$.

2.2.2. Static-flasks test

2.2.2.1. Culture medium. Distilled water (1 L) was seeded with domestic sewage supernatant (100 mL). Then, it was supplemented with carbamide ($5\,mg\,L^{-1}$) and sodium tripolyphosphate ($1.6\,mg\,L^{-1}$). Finally, culture medium was aerated for 3 days before use.

2.2.2.2. Analytical method. The concentration of the sulfide mineral flotation collectors were analysed through ultraviolet spectrophotometric method using a spectrophotometer (Shimadzu, Japan).

2.2.2.3. Test methods. Biodegradation cultures were applied in 250 mL flasks containing 90 mL of culture medium with pH of about 7.4, 10 mL of domestic sewage supernatant, 5 mg of yeast extract and proper amount of test compounds. Specifically, the initial concentration of test compounds was $10\,mg\,L^{-1}$ [17].

All experiments were carried out in a thermostated water bath at 28°C. After culturing for 7 days, 10 mL of the above mixture was transferred into another flask and cultured for 7 days, which was repeated for four periods.

Each biodegradation experiment contained triplicate cultures, together with a blank culture experiment. The biodegradation extent (D) can be expressed as follows.

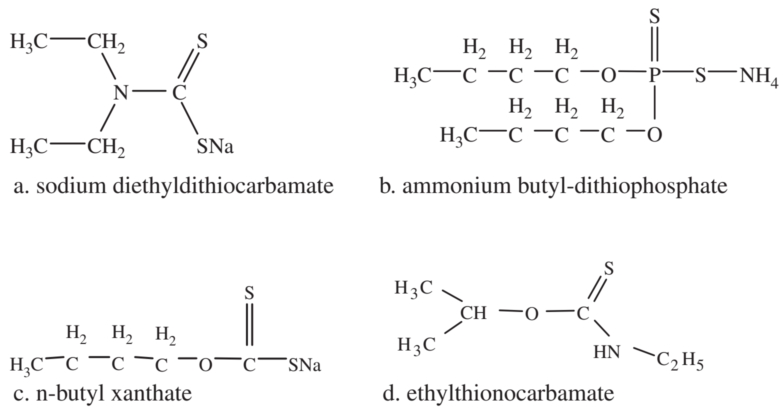

Fig. 1. Chemical structures of (a) sodium diethyldithiocarbamate, (b) ammonium butyl-dithiophosphate, (c) n-butyl xanthate and (d) ethylthionocarbamate.

$$D = \frac{[(M_0 - B_0) - (M_7 - B_7)]}{M_0 - B_0} \times 100 \quad (1)$$

where D—biodegradation extent, %, M_0 and M_7 were the concentrations of collectors measured in the culture medium at the beginning and at the end of each period, respectively.

B_0 and B_7 were the concentrations measured in the blank test at the beginning and at the end of each period, respectively.

2.2.3. OECD 301B (Modified Sturm test)

2.2.3.1. Test schematic diagram. Fig. 2 is the schematic diagram of OECD 301B. The schematic diagram including: (1) F, gas flowmeter, (2) A_1, A_2, A_3, the first, second and third NaOH absorbing bottle, respectively, (3) D_1, $Ba(OH)_2$ absorbing bottle, (4) W, pure-water wash bottle, (5) E, buffering flask, (6) T, reaction bottles, containing mineral medium, test substance and inoculum, (7) N, endogenous respiration reaction bottle, containing mineral medium and inoculum, (8) B, blank bottle, only containing mineral medium, (9) S, reference reaction bottle, containing mineral medium, reference substance and inoculum and (10) D_2, D_3, D_4 are absorbing unit of CO_2, the CO_2 produced in the process of biodegradation is absorbed.

2.2.3.2. Mineral medium. The composition of mineral medium used, with pH of about 7.4, was as follows (in $g\,L^{-1}$ distilled water): potassium dihydrogen orthophosphate (0.085), dipotassium hydrogen orthophosphate (0.2175), disodium hydrogen orthophosphate dehydrate (0.334), ammonium chloride (0.005), calcium chloride dehydrate (36.40), magnesium sulphate heptahydrate (22.50) and iron (III) chloride hexahydrate (0.25).

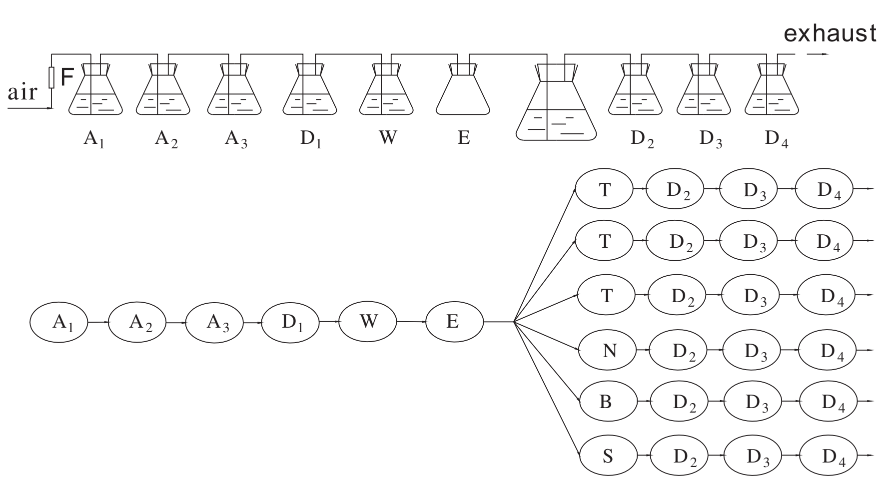

Fig. 2. The schematic diagram of OECD 301B.

2.2.3.3. Reference substances. Aniline is known as readily biodegradable and is recommended by OECD guideline 301 as reference compounds to check the reproducibility of the methods [6].

2.2.3.4. Preparation of activated sludge. Activated sludge was collected from a sewage treatment plant (Wuhan, P.R. China). It was first filtered to remove large particles and then transferred into a jar and aerated for 7 days which improved the precision of the test methods by reducing blank values. Activated sludge was characterized by its suspended solids content and viable cell count. To determine the suspended solids content, 100 mL activated sludge was filtered through a pre-weighed filter paper. The filter paper and solids were then oven-dried, cooled and reweighed [18]. Afterwards, the sludge was concentrated to 15 g L^{-1} solids. Viable cell counts were determined by plate count agar tests. A large number of live microbes were found under microscope with a cell number of about (7.0–8.5) $\times 10^7$ CFU mL^{-1}.

2.2.3.5. Test methods. Modified Sturm tests were conducted according to the OECD 301B method [6]. Characterized biomass was placed into a sealed vessel unit (SVU). The CO_2-free air production system consisted of an air compressor. The CO_2-free air was passed on to an air sparger with 1 input and 6 output channels and through PE-tubes to the SVU. There $Ba(OH)_2$ traps were connected to each SVU. The 6 reaction bottles were prepared as follows: 2,000 mL test organic solution with an initial concentration of 20 mg L^{-1} (as DOC) was added to the reaction bottle. Then, the inoculated sludge and inorganic nutrients were added. After that, the sludge was inoculated to achieve 150 g L^{-1} MLSS in the final 3 l of inoculated mixture. The experimental temperature was 28℃, pH was 7.5. Agitation was increased by stirring with a magnetic bar at about 300 rpm for 28 days.

During experiments, the analyses of CO_2 should be made in every second day until the 28th day. All substrates were analysed in triplicate, and then took the mean cumulative CO_2 production to eradicate any discrepancies.

3. Results and discussion

3.1. BOD_5/COD_{Cr} ratio

For the BOD_5/COD_{Cr} ratio test, the specific evaluation criteria are as follows [8].

If $BOD_5/COD_{Cr} \geq 0.45$ is considered as readily biodegradable, $0.30 \leq BOD_5/COD_{Cr} < 0.45$ is considered as partially biodegradable, $BOD_5/COD_{Cr} < 0.30$ is considered as poorly biodegradable. And the evaluation results of the collectors are shown in Table 1.

Table 1 shows that the BOD_5/COD_{Cr} of sodium diethyldithiocarbamate and ammonium butyl-dithiophosphate are 0.46 and 0.32, respectively. Therefore, they are considered as readily biodegradable and partially biodegradable. The BOD_5/COD_{Cr} of n-butyl xanthate and ethylthionocarbamate are less than 0.30, they are considered as poorly biodegradable. The biodegradability of these collectors follows the order of: sodium diethyldithiocarbamate > ammonium butyl-dithiophosphate > n-butyl xanthate > ethylthionocarbamate. And our previous study indicated that the electrical parameter of these collectors follow the same order listed above. These conclusions are in accordance with those obtained from our previous study that the biodegradability of sulfide mineral flotation collectors is mainly related to electrical parameter [19].

3.2. Static-flasks test

The biodegradability evaluation criteria are shown in Table 2. Biodegradation extent and evaluation results are presented in Table 3.

Table 3 shows that the biodegradation extents of the different sulfide mineral flotation collectors are different. And the order of the biodegradability of these collectors is: sodium diethyldithiocarbamate > ammonium butyl-dithiophosphate > n-butyl xanthate > ethylthionocarbamate.

3.3. OECD 301B (Modified Sturm test)

3.3.1. 10-day window and biodegradation level as evaluation indicators

The biodegradation level is expressed as a percentage of the theoretical CO_2 production based on the amount of test sulfide mineral flotation collectors added initially, namely $ThCO_2$ is calculated as follows:

$$ThCO_2 = \frac{CO_2 \text{ Produced (mg)}}{CO_2 \text{ Theoretical (mg)}} \times 100\%$$

According to OECD 301B tests, the maximum biodegradation level ($ThCO_2$ within 28 days) and ten-day window were usually taken as an biodegradability evaluation indicators [6]. And the results are summarized in Table 4.

For the aniline, about 100% $ThCO_2$ values were reached within 28 days. This indicated that the test was effective. Table 4 shows sodium diethyldithiocarbamate was readily biodegradable ($ThCO_2 > 60\%$ and

Table 1
Results of BOD_5, COD_{Cr} and BOD_5/COD_{Cr} of sulfide mineral flotation collectors

Collectors	BOD_5	COD_{Cr}	BOD_5/COD_{Cr}
Sodium diethyldithiocarbamate	77.24	167.92	0.46
Ammonium butyl-dithiophosphate	38.53	120.42	0.32
n-Butyl xanthate	25.19	119.95	0.21
Ethylthionocarbamate	18.29	130.65	0.14

Table 2
Biodegradability evaluation criteria of static-flasks test

Assessment index	Readily biodegradable	Partially biodegradable	Poorly biodegradable	Non biodegradable
D	>70%	40–70%	20–40%	<20%

Table 3
Biodegradation extent and evaluation results of sulfide mineral flotation collectors

Test substance	Biodegradation extent of each period				
	First period (%)	Second period (%)	Third period (%)	Fourth period (%)	Evaluation result
Sodium diethyldithiocarbamate	76.28	77.01	79.24	85.49	Readily biodegradable
Ammonium butyl-dithiophosphate	44.69	45.07	45.61	45.93	Partially biodegradable
n-Butyl xanthate	28.32	34.09	37.78	38.88	Poorly biodegradable
Ethylthionocarbamate	28.93	33.44	34.11	37.91	Poorly biodegradable

Table 4
Results of for the biodegradability assessment of sulfide mineral flotation collectors

Collectors	10-day window (%)	Maximum biodegradation level (%)	Biodegradability
Sodium diethyldithiocarbamate	86.11	88.49	Readily biodegradable
Ammonium butyl-dithiophosphate	35.86	36.17	Partially biodegradable
n-Butyl xanthate	−14.10	5.80	Poorly biodegradable
Ethylthionocarbamate	−45.39	−24.47	Poorly biodegradable (inhibitive factor)

Note: Minus means the amount of carbon dioxide from collector less than endogenous respiration.

ten-day window > 10%), ammonium butyl-dithiophosphate can be considered as partially biodegradable ($ThCO_2 < 60\%$ but 10-day window > 10%), while the n-butyl xanthate was well below the pass value, it can be considered as poorly biodegradable collectors (both $ThCO_2$ and 10-day window < 10%), it is considered relatively resistant to biodegradation, and its biodegradation can be performed with preadapted bacteria, but remains limited by the substrate inhibition [20]. Concerning ethylthionocarbamate, no biodegradation occurred and with mixed bacterial populations appeared to be inhibited.

3.3.2. IB as an evaluation indicator (Improvement on the OECD 301B tests' criterion)

The biodegradability of organic compounds can be described by three typical curves of carbon dioxide production vs. time, known as PCD curves (Fig. 3).

Fig. 3 shows that for different organic compounds, the PCD curves are different. The curve I shows that the organic can be degraded by mixed microbes without acclimation, the biodegradation rate is higher, and the area of CO_2 evolution curve is larger. The curve II represents the organic that has potential inhibitive effects on the growth of mixed

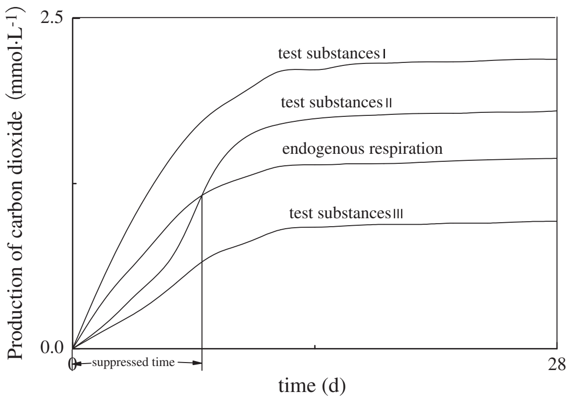

Fig. 3. Typical PCD curves for describing biodegradation of organic compounds.

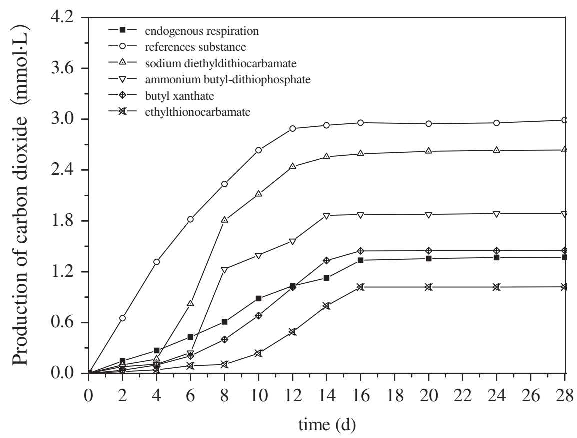

Fig. 4. The mean cumulative CO_2 production of the sulfide mineral flotation collectors.

microbial populations and the acclimation is needed. The biodegradation rate is slow, and the area of CO_2 evolution curve is lesser. The curve III is consistently situated below the PCD curve of endogenous respiration, which indicates that the organic compound is considered relatively resistant to biodegradation and toxic to the growth of microbial populations. Consequently, the area of the CO_2 evolution curve can indicate the biodegradability of organic compounds, using the production of carbon dioxide as biodegradability indicator, which is not affected by microbial cell adsorption and nitrification. Furthermore, from environmental pollution perspective, the organic compounds break down into carbon dioxide and water, which is most thorough and meaningful. So, the index of biodegradability (IB) can illustrate biodegradability of organic compounds.

$$\mathrm{IB} = \frac{A_S}{A_0} \times 100 \quad (2)$$

where A_s shows the area of the CO_2 evolution curve, A_0 is the area of the CO_2 evolution due to endogenous respiration. If $IB \geq 200$ is considered as readily biodegradable, $100 \leq IB < 200$ is considered as partially biodegradable, $IB < 100$ is considered as poorly biodegradable.

The PCD curves of sulfide mineral flotation collectors are depicted in Fig. 4. And the results of assessment are summarized in Table 5.

As shown in Fig. 4, the reference substance aniline could be degraded rapidly, IB was 253.0338, much more than 200. This indicated that aniline is readily biodegradable, microbial activity occurred effectively and the test was effective. At the beginning of the test, the PCD curves of sodium diethyldithiocarbamate, ammonium butyl-dithiophosphate and n-butyl xanthate are located below the endogenous respiration, indicating that they have a potential toxicity, and have some inhibitory effect on mixed bacterial populations. However, after a few of days of acclimation, the cumulative evolution amount of CO_2 resulting from the metabolism of sodium diethyldithiocarbamate, ammonium butyl-dithiophosphate and n-butyl xanthate gradually becomes over the cumulative evolution amount of CO_2 resulting from endogenous respiration. As shown in Fig. 4, the suppressed time of different collectors are comparatively different, and the suppressed time of sodium diethyldi-

Table 5
Classification for biodegradability of sulfide mineral flotation collectors

Test substance	The area	IB (%)	Biodegradability
Endogenous respiration	26.6347	–	–
Aniline	67.3948	253.0338	Readily biodegradable
Sodium diethyldithiocarbamate	54.0649	202.9867	Readily biodegradable
Ammonium butyl-dithiophosphate	37.4315	140.5366	Partially biodegradable
n-Butyl xanthate	26.3681	99.0013	Poorly biodegradable
Ethylthionocarbamate	16.8247	63.1683	Poorly biodegradable

thiocarbamate is the shortest, followed by ammonium butyl-dithiophosphate, and n-butyl xanthate is the longest, the suppressed time attained 4, 7, 12 days, respectively. The area of the CO_2 evolution produced by endogenous respiration is 26.6347, the area of CO_2 evolution produced from sodium diethyldithiocarbamate, ammonium butyl-dithiophosphate and n-butyl xanthate is 54.0649, 37.4315 and 26.3681, along with the IB can be calculated as 202.9867, 140.5366 and 99.0013, respectively. The test result indicates that sodium diethyldithiocarbamate is considered as readily biodegradable, ammonium butyl-dithiophosphate is partially biodegradable and n-butyl xanthate is poorly biodegradable. However, the PCD curve of ethylthionocarbamate is consistently located below the PCD curve of endogenous respiration, indicating that ethylthionocarbamate is toxic to the growth of mixed microbial populations, inhibiting the microbial populations' activity, the IB is only 63.1683, much less than 100, therefore, so it can be considered as poorly biodegradable sulfide mineral flotation collector.

3.4. Comparison of different biodegradability tests

Tables 1 and 3–5 show that the consistent conclusions have been obtained through BOD_5/COD_{Cr} ratio test, Static-flasks test and OECD 301B biodegradability tests. Namely, sodium diethyldithiocarbamate is considered as readily biodegradable and ammonium butyl-dithiophosphate is considered as partially biodegradable collector. However, n-butyl xanthate and ethylthionocarbamate can be considered as poorly biodegradable collectors. These results have some differences with the previous study [21], namely, the biodegradability of sulfide mineral flotation collectors according to previous study were slightly superior to those three biodegradability tests. Especially, the primary biodegradation extent of n-butyl xanthate can reach 93.70% according to previous study, but in the present investigation, n-butyl xanthate is considered as poorly biodegradable collector. The main reason for those differences is that a small amount of yeast extract was added with co-metabolism substrate during the biodegradation of sulfide mineral flotation collectors according to "Water Quality-Evaluation in an Aqueous Medium of the Aerobic Biodegradability of Organic Compounds" biodegradability test. It can greatly enhance the biodegradability of sulfide mineral flotation collectors. Furthermore, the results of biodegradability tests are very dependent on the quantity and quality of the biomass has been reported [22, 23]. Therefore, minor differences in the extent of biodegradability were observed. The aerobic biodegradability follows the order: sodium diethyldithiocarbamate > ammonium butyl-dithiophosphate > n-butyl xanthate > ethylthionocarbamate. The results are in agreement with those reported in my previous study [19,21] that: $k_{\text{sodium diethyldithiocarbamate}} > k_{\text{ammonium butyl-dithiophosphate}} > k_{\text{n-butyl xanthate}} > k_{\text{ethylthionocarbamate}}$. The main reason for this is that with different structures, the biodegradability of sulfide mineral flotation collectors is comparatively different. And according to our previous findings, the biodegradability of sulfide mineral flotation collectors is mainly related to electrical parameter, such as electronic energy (EE), total energy (TE) and the energy of the lowest unoccupied molecular orbital (E_{LUMO}). Namely, the biodegradation rate of sulfide mineral flotation collectors is significantly affected by the rate of the key enzyme-catalyzed reactions. However, steric parameter and hydrophobic parameter have a small impact on biodegradability of sulfide mineral flotation collectors [19].

4. Conclusions

The aerobic biodegradability of the typical sulfide mineral flotation collectors, namely sodium diethyldithiocarbamate, ammonium butyl-dithiophosphate, n-butyl xanthate and ethylthionocarbamate, was evaluated according to the integrated assessment method including three biodegradability tests. Consistent conclusions have been obtained through these three biodegradability tests. Namely, sodium diethyldithiocarbamate is a readily biodegradable collector and ammonium butyl-dithiophosphate is partially biodegradable. However, n-butyl xanthate and ethylthionocarbamate are considered as poorly biodegradable collectors. The aerobic biodegradability follows this order: sodium diethyldithiocarbamate > ammonium butyl-dithiophosphate > n-butyl xanthate > ethylthionocarbamate.

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