Fire extinguishing properties of novel ferrocene/surfynol 465 dispersions

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Abstract

This paper reports a novel fire-extinguishing agent: an aqueous dispersion of fine ferrocene particles. In this study, the ferrocene–water–surfactant dispersions were prepared by optimizing the gas-phase concentration of ferrocene, and their ability to extinguish heptane fires was examined. The fire-extinguishing efficiency was characterized by three parameters: the ferrocene concentration in the dispersion (0–175 ppm), the surfactant used, and the ferrocene particle size (d_{50} = 10.4, 11.4, 21.5, and 68.8 μm). The results indicated that (1) the ferrocene (d_{50} = 10.4 mm)–water–surfynol 465 dispersion is the most stable among the dispersions tested, (2) the ferrocene–water–surfynol 465 dispersions have an optimal value of ferrocene concentration regarding the extinguishing time, and (3) in the ferrocene particle size range of 10.4–21.5 μm, the minimum extinguishing time of the ferrocene–water–surfynol 465 dispersions is remarkably shorter (1.2 sec) than those observed when using a conventional wet chemical agent (45 wt% aqueous solution of potassium carbonate, 12.9 s).

Keywords: Ferrocene, Fire-extinguishing agent, Dispersion, Extinguishing time, Surfynol 465

1. Introduction

Accidental deaths due to residential fires pose a social problem in many countries [1–4]; thus, more effective fire-extinguishing agents are required for preventing loss of life. Typical fire-extinguishing agents include water mist (with additives) [5], foams [6], inert gases [7], and inorganic salts, such as bicarbonates and phosphates [8]. Some studies have reported that transition metals act as a radical scavenger in a flame, thereby producing a fire suppression effect. Iron pentacarbonyl [9, 10], cuprous chloride [11], and manganese dichloride [12] are known to function as flame inhibitors. For instance, iron pentacarbonyl easily produces fire-inhibiting iron species, such as Fe, FeO, FeO_2, FeOH, and $Fe(OH)_2$, in a flame, thus exhibiting high flame-suppression efficiency [13]. However, these transition metal compounds are highly toxic, which restricts their practical application as fire-extinguishing agents. In contrast, ferrocene ($FeCp_2$, see Fig. 1a), which is also a transition metal compound, is less toxic than iron petacarbonyl. Linteris et al. demonstrated that ferrocene has an exceptional ability to reduce the burning velocity of premixed CH_4 flames [13]. We have previously shown that the concentration of the inhibitor required to extinguish Class A fires decreases by a factor of 11 when ferrocene is used instead of ammonium dihydrogen phosphate, which is the main component of the extinguishing agent “ABC dry chemical” [14]. However, the study also revealed that higher ferrocene fractions cannot extinguish flames. When inhibiting species (e.g., Fe, FeOH, $Fe(OH)_2$, and iron oxides) are present in excess of the super-equilibrium

concentrations, the gas-phase catalytic recombination of radicals in a flame is found to be effective. However, at higher concentrations, the effective flame inhibition is not observed because of the condensation of the active inhibiting species in the gas phase [15]. Therefore, when employing ferrocene as a fire-extinguishing agent, the concentration of ferrocene must be optimized for flame inhibition.

The present study proposes a novel fire-extinguishing agent: an aqueous dispersion of fine ferrocene particles. The advantage of using aqueous dispersions includes the easy preparation of dispersions with optimum concentration of ferrocene. In this study, ferrocene powder was dispersed in water using a surfactant, and the influence of surfactants on the dispersion stability was evaluated by visual observation and turbidity measurements. Subsequently, the effects of ferrocene concentration and particle size on the extinguishing time were experimentally investigated. In addition, this study also compares the extinguishing time of the ferrocene–water dispersion with that of a 45 wt% aqueous solution of potassium carbonate (K_2CO_3), which is a conventional wet chemical extinguishing agent.

2. Material and methods

2.1 Chemicals

Ferrocene (>98.0%, Wako Pure Chemical Industries, Ltd., Japan) and potassium carbonate

(>99.5%, Junsei Chemical Co., Ltd, Japan) were used as received. *n*-Heptane (>99.9%, Kanto Chemical Co. Inc., Japan) was treated over molecular sieves to remove the trace amount of water. Water used in this study was deionized (<1 μS cm^{-1}). Surfynol 465 (Air Products and Chemicals, Inc., USA), and *n*-dodecyltrimethylammonium chloride (DTAC, Kanto Chemical Co. Inc., Japan) were used as nonionic and cationic surfactants, respectively (see Fig. 1). The latter surfactant was selected, because it is commonly used. Surfynol 465, a nonionic *gemini* surfactant, is a less toxic, new-generation amphiphile with two interlinked surfactant molecules [16]. Gemini surfactants are also referred as dimeric-, twin-, or bis-surfactants. They are highly efficient at reducing surface tension [17] and are also known to have very low critical micelle concentrations (CMCs) [18]. Surfynol 465 complies with Environmental Protection Agency and Food and Drug Administration regulations.

In the present study, the CMCs of the surfactant were determined by the du Noüy ring method [17].

2.2 Preparation of aqueous dispersions of ferrocene

Ferrocene powder was ground in a planetary mill (Pulverisette 7, Fritsch, Germany) using 2-mm-diameter zirconia balls or crushed in an agate mortar to produce four ferrocene samples with micron-sized particles (S1–S4). The particle size distributions of these samples were measured by a

laser diffraction particle size analyzer (SALD 7000, Shimadzu Corp., Japan) and microscopic observation (DMI 3000B, Leica, Germany). The particle size analyses provided d_{50} values of 10.4, 11.4, 21.5, and 68.8 μm for the four samples (S1–S4, respectively); the larger the sample number, the greater the median diameter. As an example, the particle size distribution of S2 is shown in Fig. 2.

Milled ferrocene powder was first added to deionized water to obtain a dispersion followed by the addition of the surfactant. To ensure micelle formation, the surfactants were concentrated to twice the CMC value. Subsequently, the aqueous dispersions of ferrocene were prepared by ultrasonication (43 kHz) for 20 min at 50 °C. For experiments with surfynol 465, the dispersion temperature was maintained below its cloud point to prevent the separation of the aqueous media into two phases. The cloud point of surfynol 465 was determined according to a standard test method [19].

2.3 Dispersion stability of the aqueous dispersions of ferrocene

In general, the turbidity of dispersion is positively correlated with the amount of suspended particles in that dispersion; in other words, increasing the amount of dispersed ferrocene particles increases the turbidity of the dispersion. The stabilities of the aqueous dispersions of ferrocene (100 ppm (mass/mass)) were evaluated by visual observation and by measuring the turbidity (NTU for

formazine) with a turbidimeter (2100Q, Hach Co., USA) at room temperature. The turbidimeter was calibrated using 20, 100, and 800 NTU *StablCal* standards (Hach Co., USA). Turbidity was continuously measured 12 times for each sample. To negate the color of the sample, the data were determined from a ratio of 90° scattered light signals to transmitted light signals.

2.4 Extinguishment experiments

Fig. 3 illustrates the experimental apparatus. A pan with 83-mm-diameter was employed for *n*-heptane pool fire burning. After pre-burning for 20 s at a steady burning condition, the aqueous dispersion of ferrocene or 45 wt% K_2CO_3 *aq*. was discharged at a flow rate of 250 mL min^{-1} for 45 s. The distance between the nozzle and the pan was set to 600 mm. The spray angle was approximately 60°.

Typical methods for measuring droplet size include laser grating [20], laser diffraction [21], phase Doppler [22], photographic [23], and immersion techniques [24]. The immersion method employed in this study involves collecting droplets using an immiscible liquid, and then determining the droplet size by microscopic observation (DMI 3000B, Leica, Germany). Notably, the determined droplet diameters may sometimes vary with the measuring method used [24]. Nevertheless, on the basis of the immersion method using silicone oil, the Sauter mean diameter ($d_{32} = \sum (n_i d_i^3) / \sum (n_i d_i^2)$), volume median diameter (d_{50}), and d_{99} of the pure water mist droplets collected at 600 mm

below the nozzle were determined to be 313, 207, and 480 μm, respectively.

In this experiment, the extinguishing time was measured for the samples S1–S4 over the ferrocene concentration range of 0–175 ppm. Each measurement was repeated 10 times to confirm the reproducibility.

3. Results and discussion

3.1 Dispersion stability of the aqueous dispersions of ferrocene

At a dispersion temperature of 30 °C, a ferrocene precipitate was immediately produced. To avoid further unstable dispersions, the dispersion temperature was hereafter increased to 50 °C.

Figs. 4 and 5 show the photographs of the S1–S4 ferrocene/surfynol 465 dispersions. In the photographs presented in Fig. 4, the dispersions of S1 ferrocene are in the left test tubes, whereas the right tests tubes are filled with the dispersions containing S2 ferrocene. In Fig. 5, the dispersions of S3 ferrocene and S4 ferrocene are in the left and right test tubes, respectively. Dispersions of S1 or S2 ferrocene were initially yellow and turbid, whereas the dispersions containing S3 or S4 ferrocene yielded a small amount of yellow precipitate (i.e., ferrocene powder), even at zero min. After 120 min, yellow precipitates were observed at the bottoms of the test tubes containing the S3 or S4 dispersions. Conversely, the S1 and S2 dispersions produced little precipitate and appeared cloudier than those containing S3 or S4 ferrocene after 120 min. These results suggest that the stability of the

dispersion depends strongly on the particle size of ferrocene; the dispersion stability decreased in the following order: S1 > S2 > S3 > S4.

To quantitatively evaluate the settling behavior, turbidity was measured using the turbidimeter for S1–S4 ferrocene/surfynol 465 and S1 ferrocene/DTAC dispersions. Fig. 6 plots turbidity against time; the results indicate the same trend for stability as reported above on the basis of visual observation. The results, obtained from the visual observations and turbidity measurements, simultaneously suggest a negative correlation between ferrocene particle size and dispersion stability. The turbidity of the S1 ferrocene/surfynol 465 dispersion was higher than that of the S1 ferrocene/DTAC dispersion, implying surfactant-dependent stability of the dispersion.

Unfortunately, the present work was unable to elucidate the reason behind better efficiency of the nonionic surfactant in dispersing the fine ferrocene particles compared with the cationic surfactant; further work is needed to understand such behavior of the surfactants.

3.2 Fire suppression experiments

Table 1 lists the extinguishing times of the aqueous dispersions containing S1, S2, S3, and S4 ferrocene/surfynol 465, S1 ferrocene/DTAC for 100 ppm ferrocene concentration, and 45 wt% K_2CO_3 aq. The tests conducted without ferrocene confirmed that pure water mist and the aqueous solutions of surfynol 465 or DTAC at concentrations of two- and five-times greater than the CMC

are unable to extinguish the heptane fire, allowing a direct evaluation of the suppression ability of ferrocene. We found the extinguishing time of ≤8 s for all the four studied aqueous dispersions of ferrocene, whereas 45 wt% K_2CO_3 aq. took 12.9 s (*SD* = 5.9 s) to extinguish the heptane fire. This indicates that these aqueous dispersions of ferrocene are significantly more effective than the conventional extinguishing agents. The extinguishing times of the three of the four studied dispersions of ferrocene are as follows: 1.2 s (*SD* = 0.4 s) for S1, 1.2 s (*SD* = 0.3 s) for S2, and 1.2 s (*SD* = 0.4 s) for S3. Moreover, these dispersions (S1–S3) exhibited remarkably smaller standard deviations compared with the conventional extinguishing agents. In contrast, the aqueous dispersion of S4 ferrocene exhibited an extinguishing time of 7.7 s (*SD* = 3.2 s). No significant difference was observed in terms of extinguishing time between S1 ferrocene/surfynol 465 and S1 ferrocene/DTAC dispersions at 100 ppm ferrocene concentration.

To elucidate the effects of ferrocene concentration and particle size on the extinguishing time, fire suppression experiments were conducted at ferrocene concentrations ranging from 0 ppm to 200 ppm. Fig. 7a plots the extinguishing time as a function of the S4 ferrocene concentration. The error bars represent one standard deviation. This figure clearly shows that the dispersion fails to extinguish the pool fire at ferrocene concentrations below 50 ppm and above 175 ppm; the higher limit is attributed to the condensation of active inhibiting species in the flame [15]. This experiment also revealed that the extinguishing time reaches a minimum value at 100 ppm ferrocene

concentration with the smallest standard deviation. This is in good agreement with previous results [14], indicating fire suppressing ability of aqueous dispersions of ferrocene at an optimum ferrocene concentration.

The extinguishing times of the S1–S4 ferrocene/surfynol 465 and S1 ferrocene/DTAC dispersions are depicted in Fig. 7b. Although the curves themselves have individual curvatures, they all show a similar *U*-shaped curve. The extinguishing times of the S1 and S2 ferrocene/surfynol 465 dispersions were found to be <3 s with a minimum value of approximately 1 s (*SD* = 0.3 s). The extinguishing time of the S3 ferrocene/surfynol 465 dispersion reached a minimum value (1.2 s, *SD* = 0.3 s) at 100 ppm ferrocene, although this dispersion exhibited larger extinguishing times and standard deviations at lower and higher ferrocene concentrations when compared with the S1 and S2 ferrocene/surfynol 465 and S1 ferrocene/DTAC dispersions. We confirmed that all the studied dispersions failed to extinguish the heptane fire at 5 ppm ferrocene; no stable dispersions were obtained when the ferrocene concentration exceeded 200 ppm.

Fire-extinguishing efficiency is impacted by chemical suppression effect and/or physical effect. The latter involves the cooling of flame and fuel surface, the exclusion of oxygen, and radiation attenuation [25]. The difference between physical effects on the extinguishing ability of the liquids with and without ferrocene was negligible probably because of the following facts: (1) the surfactant concentrations, which significantly affect droplet size, were equivalent and (2) no heptane pool fires

were extinguished by the aqueous solution of Surfynol 465 at concentration five-times greater than the CMC. In general, the spray properties are mainly affected by droplet size and droplet momentum [26], and the droplet size primarily depends on specific gravity, viscosity, surface tension, pressure, and temperature [27]. Among these variables, the latter three were expected to be constant in this study. The former two had a limited impact on the spray properties as the aqueous dispersions contained very low concentrations of ferrocene (i.e., 100 ppm). Moreover, the particle diameter of ferrocene powder was approximately one order of magnitude smaller than that of the spray droplet. All these facts together imply that the presence of ferrocene particles does not significantly influence the spray properties. Hence, these experiments allowed us to confirm the effectiveness of the ferrocene dispersions in extinguishing fire.

These suppression experiments revealed a negative relation between ferrocene particle size and dispersion stability. In addition, the particle size was also found to be negatively correlated with the extinguishing time. This result agrees well with the fact that the particle size of suppressants generally governs their fire-extinguishing ability [28]; also, it suggests that the extinguishing time and dispersion stability might be improved by using smaller ferrocene particles. However, a comparison between the extinguishing times of S1 ferrocene/surfynol 465 and S1 ferrocene/DTAC dispersions demonstrated that the surfactant had little influence on extinguishing time in this study; furthermore, systematically investigating the influence of the surfactant on the extinction ability

using a large variety of surfactants is necessary.

4. The further advantages of the aqueous dispersions of ferrocene

In this study, high-performance dispersions were easily prepared via ultrasonication using surfactants including surfynol 465, an ethoxylated acetylenic diol consisting of carbon, hydrogen, and oxygen atoms. The aqueous dispersion of ferrocene with such surfactants may also derive secondary benefits such as being free of halogens and phosphorous. The absence of halogens makes the dispersion more environment friendly. In addition, ammonium dihydrogen phosphate has been widely used as a multipurpose fire-extinguishing agent; however, the price of phosphate rock fluctuates dramatically (e.g., the price peaked in 2008) [29]. Thus, free of phosphorous can avoid exposure to volatile phosphate prices.

Further work is required to understand the chemical stability of ferrocene in water as both chemical and dispersion stabilities are necessary for practical use of aqueous dispersions of ferrocene.

5. Conclusions

Novel aqueous dispersions of fine ferrocene particles were developed to optimize the ferrocene concentration required to extinguish a flame. The ability of these aqueous dispersions of ferrocene to

extinguish heptane pool fires was also evaluated. This investigation highlighted the following results:

1. Using a variety of surfactants (nonionic surfynol 465and cationic DTAC), ultrasonication for 20 min at 50 °C easily allowed the preparation of aqueous dispersions of milled ferrocene powder with mean diameters of 10.4 μm (S1), 11.4 μm (S2), 21.5 μm (S3), and 68.8 μm (S4).
2. Visual observations and turbidity measurements clearly demonstrated that ferrocene particle size was negatively correlated with dispersion stability.
3. The average extinguishing times of S1–S3 ferrocene–water–surfynol 465 at 100 ppm ferrocene, S1 ferrocene–water–DTAC, and 45 wt% K_2CO_3 *aq*. were 1.2 s, 1.0 s, and 12.9 s, respectively, indicating greater efficiency of fire suppression of aqueous dispersions of ferrocene compared with the conventional extinguishing agents.
4. In the ferrocene concentration range of 0 to 175 ppm, the extinguishing time was found to be negatively correlated with the ferrocene particle size.

On the basis of the results described above, we conclude that the novel, phosphorous- and halogen-free aqueous dispersion of ferrocene possesses remarkable fire-extinguishing efficiency.

Acknowledgments

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Figure captions

Fig. 1 Chemical structures of (a) ferrocene, (b) surfynol 465 ($m + n = 10$), and (c) *n*-dodecyltrimethylammonium chloride (DTAC).

Fig. 2 Particle size distribution of S2 ferrocene powder.

Fig. 3 Schematic of the experimental apparatus for the extinguishing test.

Fig. 4 Appearance of the aqueous dispersions containing surfynol 465 after (a) 0 min, (b) 60 min, (c) 120 min, and (d) 240 min (left test tubes: aqueous dispersions of S1 ferrocene; right test tubes: aqueous dispersions of S2 ferrocene).

Fig. 5 Appearance of aqueous dispersions containing surfynol 465 after (a) 0 min, (b) 60 min, (c) 120 min, and (d) 240 min (left test tubes: aqueous dispersions of S3 ferrocene; right test tubes: aqueous dispersions of S4 ferrocene).

Fig. 6 Turbidity of aqueous dispersions of ferrocene as a function of time (triangles = S1 ferrocene–water–surfynol 465, squares = S2 ferrocene–water–surfynol 465, lozenge = S3

ferrocene–water–surfynol 465, circles = S4 ferrocene–water–surfynol 465, and stars = S1 ferrocene–water–DTAC).

Fig. 7 (a) Extinguishing time versus concentration of S4 ferrocene and (b) extinguishing times of the suppressants used in this study versus ferrocene concentration (triangles = S1 ferrocene–water–surfynol 465; squares = S2 ferrocene–water–surfynol 465; lozenge = S3 ferrocene–water–surfynol 465; circles = S4 ferrocene–water–surfynol 465; and stars = S1 ferrocene–water–DTAC. The ferrocene concentration was 100 ppm). The error bars represent the standard deviation.

Table caption

Table 1 Extinguishing times and standard deviations of the suppressants used in this study.

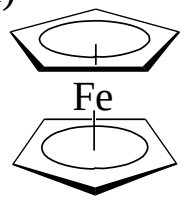
Table 1

Suppressant	Extinguishing time (s)	Standard deviation (s)
S1 ferrocene [a]/surfynol 465	1.2	0.4
S2 ferrocene [a]/surfynol 465	1.2	0.3
S3 ferrocene [a]/surfynol 465	1.2	0.4
S4 ferrocene [a]/surfynol 465	7.7	3.2
S1 ferrocene [a]/DTAC	1.0	0.1
45 wt.% K_2CO_3 *aq*.	12.9	5.9

a: Ferrocene concentration was set to 100 ppm.

Figure 1

(a)



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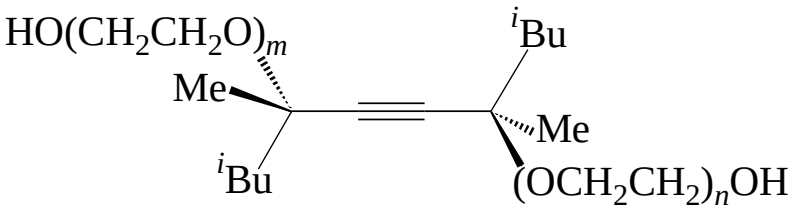

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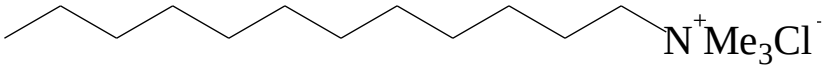


Figure 2

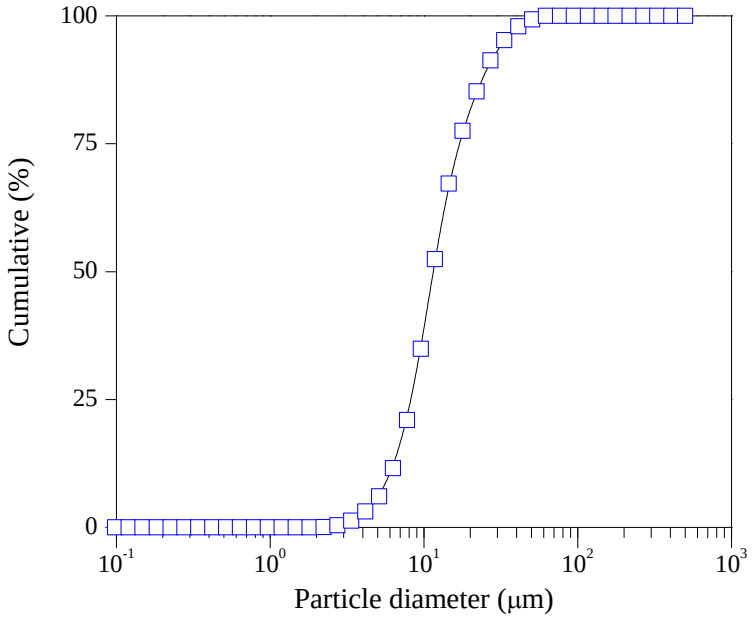

100
75
50
25
0
Cumulative (%)
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
Particle diameter (μm)

Figure 3

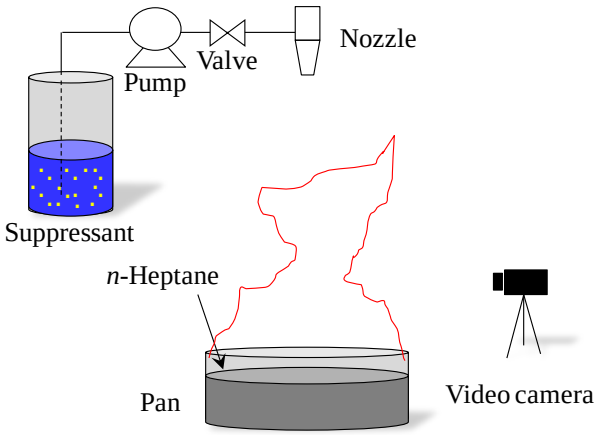


Figure 4

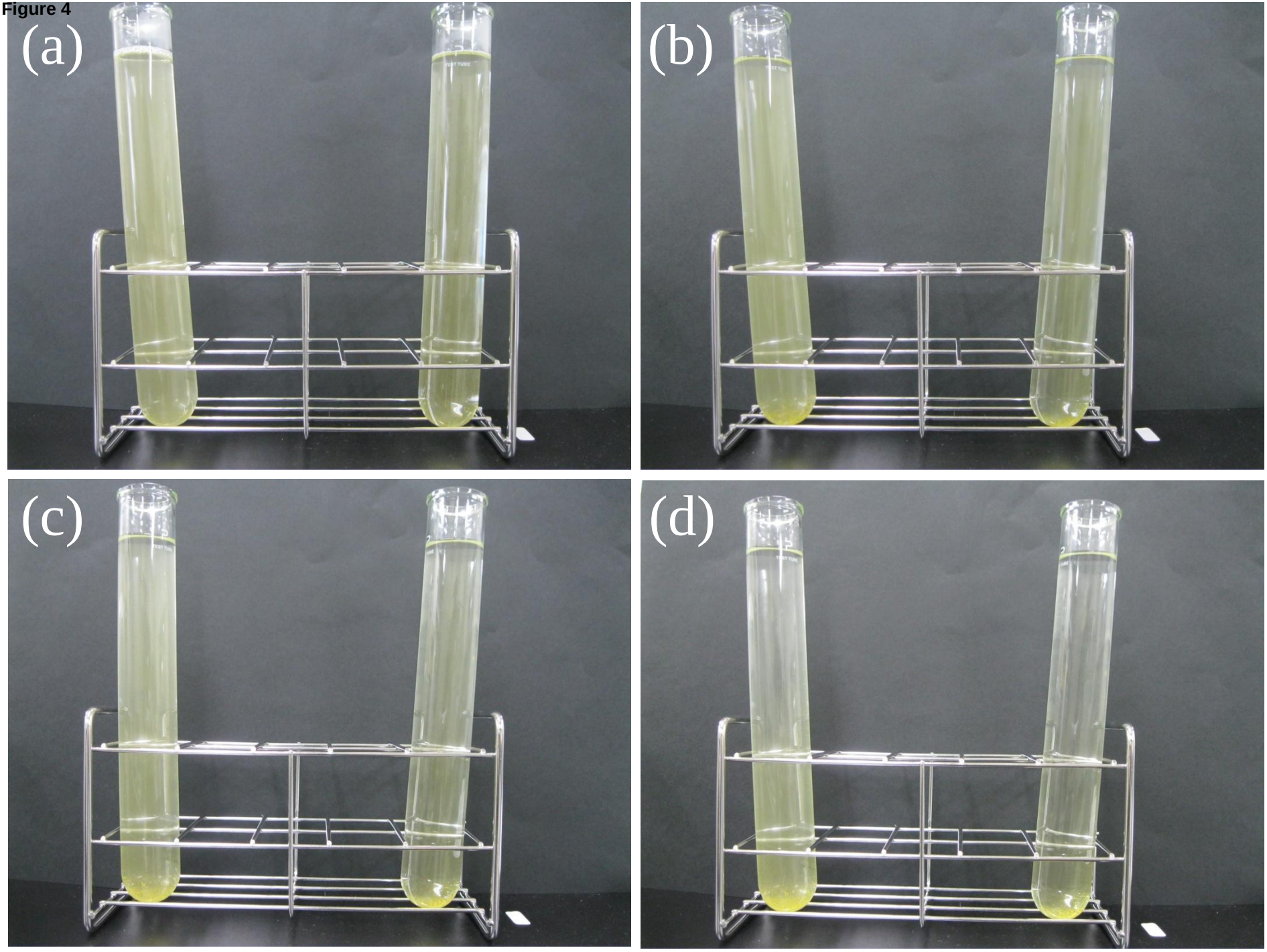


Figure 5

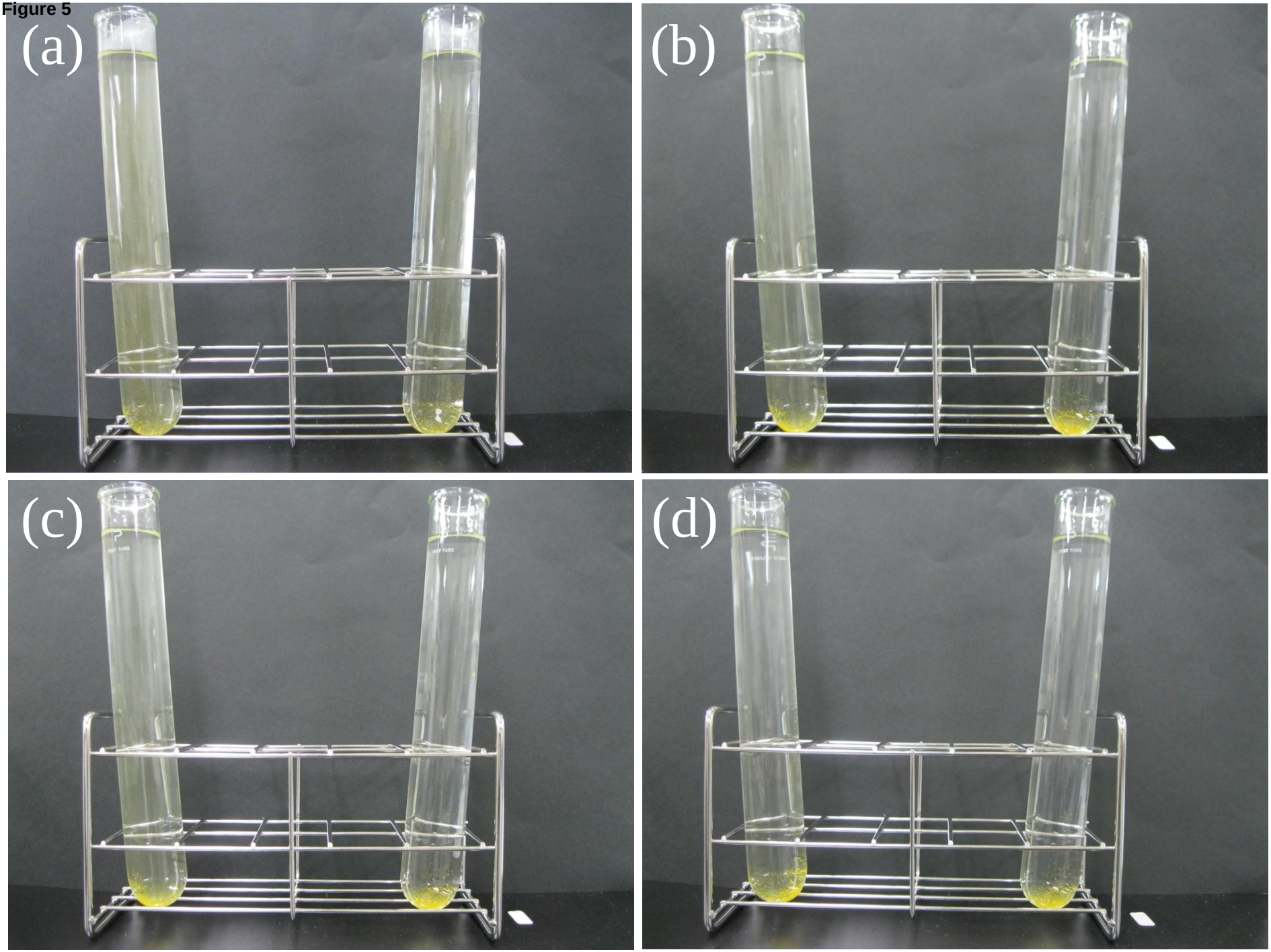


Figure 6

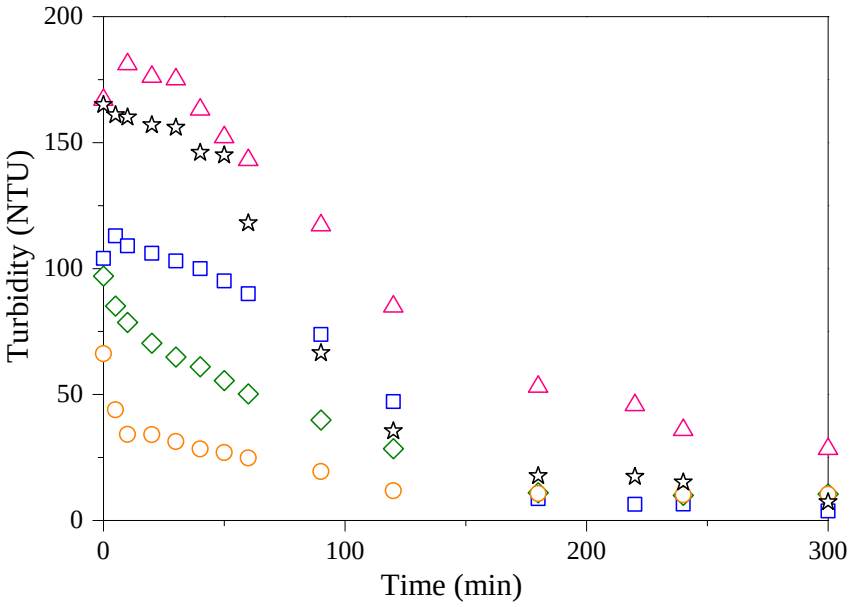
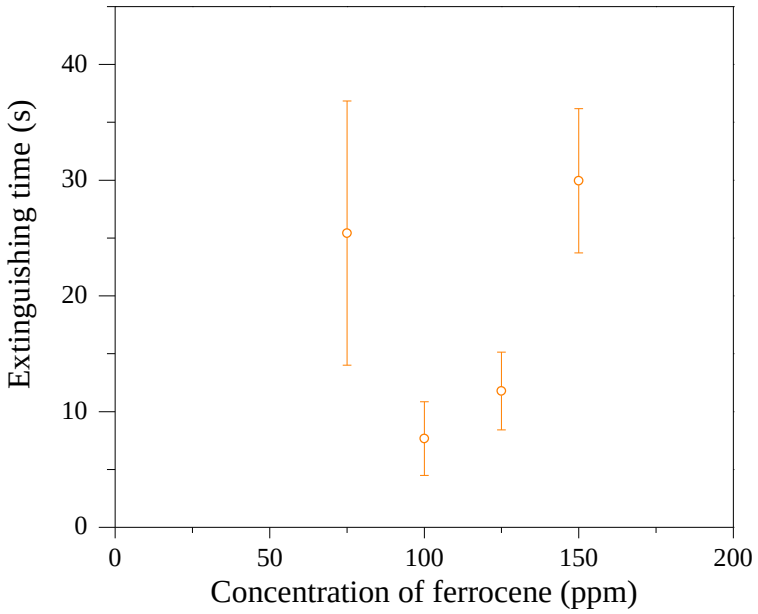
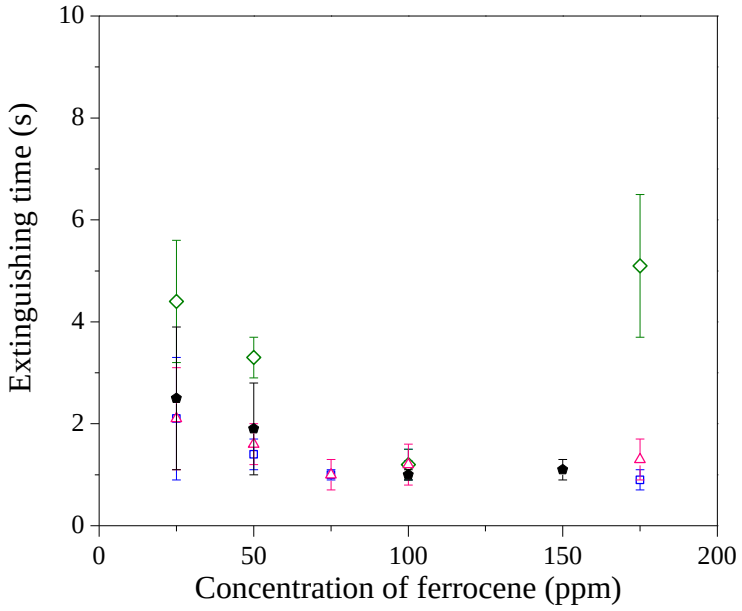


Figure 7

(a)


Extinguishing time (s)
0
10
20
30
40
0
50
100
150
200
Concentration of ferrocene (ppm)

(b)


Extinguishing time (s)
0
2
4
6
8
10
0
50
100
150
200
Concentration of ferrocene (ppm)