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Hg^0 absorption in potassium persulfate solution*

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Abstract: The aqueous phase oxidation of gaseous elemental mercury (Hg^0) by potassium persulfate (KPS) catalyzed by Ag^+ was investigated using a glass bubble column reactor. Concentration of gaseous mercury and potassium persulfate were measured by cold vapor atom absorption (CVAA) and ion chromatograph (IC), respectively. The effects of pH value, concentration of potassium persulfate and silver nitrate (SN), temperature, Hg^0 concentration in the reactor inlet and tertiary butanol (TBA), free radical scavenger, on the removal efficiency of Hg^0 were studied. The results showed that the removal efficiency of Hg^0 increased with increasing concentration of potassium persulfate and silver nitrate, while temperature and TBA were negatively effective. Furthermore, the removal efficiency of Hg^0 was much better in neutral solution than in both acidic and alkaline solution. But the influence of pH was almost eliminated by adding $AgNO_3$. High Hg^0 concentration has positive effect. The possible reaction mechanism of gaseous mercury was also discussed.

Key words: Potassium persulfate, Gaseous elemental mercury, Absorption, Reaction mechanism
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INTRODUCTION

Lipfert *et al.*(1995) reported that methylmercury level in edible fish doubled near a 1000 MW power plant. Mercury is a leading concern among the air toxic metals addressed in the 1990 Clean Air Act Amendments (CAAA) because of its volatility, persistence, and bioaccumulation as methylmercury in the environment and its neurological health impacts. Coal-fired utility boilers are now identified as the largest source of mercury in the United States (US EPA, 1997; 1998), releasing approximately 50 t of mercury annually or about one-third of the total anthropogenic emission. The concentration of mercury from coal-fired flue gases was at the level of several tens microgrammes per cubic meter.

Hg^0 absorption rate is mainly controlled by liquid phase resistance due to its very low solubility in water. So, it is necessary to use oxidizers to remove gaseous Hg^0 by transforming it into Hg^{2+}. The standard potential of the redox-pair Hg^0/Hg^{2+} is 0.85 V. To achieve complete reaction, the over-potential should be 0.3~0.4 V. Therefore, the standard oxidizing potential of the oxidizers applied should at least be 1.2 V (van der Vaart *et al.*, 2001). Some oxidizers meeting the above criteria are listed as follows: $KMnO_4$, $K_2S_2O_8$ ($AgNO_3$), K_2CrO_7, H_2O_2, $HgCl_2$, $NaClO_4$, $NaClO_3$ or Cl_2 (van der Vaart *et al.*, 2001; Morita *et al.*, 1983; Zhao and Rochelle, 1996; 1998; 1999; House, 1962). The standard oxidizing potential of potassium persulfate (KPS) is 2.01 V, which is higher than that of potassium permanganate (1.7 V). The general catalysts found in the literature include copper and silver ions (House, 1962), manganese (House, 1962; Lenka and Dash, 1983; Kislenko *et al.*, 1997), cerium (House, 1962; Skarzewski, 1984), and cobalt (Lenka and Dash, 1983). Liang *et al.*(2004a; 2004b) studied persulfate oxidation in situ remediation of trichloroethylene (TCE) catalyzed by ferrous ion.

Little information on the mechanism of the reaction of Hg^0 and potassium persulfate is available in the literature. In this study, the feasibility of Hg^0 absorption in potassium persulfate catalyzed by silver

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ion was evaluated in a bubble column reactor. This study was aimed at determining the removal efficiency of Hg^0 in solution under different conditions. Besides, the mechanism of the reaction between Hg^0 and potassium persulfate in the process was also studied.

EXPERIMENTAL METHODS

Chemicals

Mercury permeation tube (USA VICI Metronics), potassium persulfate (>99.5%, AR), hydroxylamine hydrochloride (>98.5%, AR), nitric acid (GR), hydrochloric acid (GR), sulfuric acid (GR), sodium hydroxide (>98.0%, GR), stannous chloride (>98.0%, CP), stannum (>99.9%, AR), potassium permanganate (>99.5%, GR), potassium dichromate (>99.8%, GR), mercury chloride (>99.0%, AR), silver nitrate (>99.8%, AR), anhydrous sodium carbonate (>99.8%, AR), sodium bicarbonate (>99.5%, AR), acetone (>99.5%, AR), tertiary butanol (CP). These reagents were purchased from Shanghai Chemical Reagents Company.

Batch experiment procedures

All batch experiments were performed in 500 ml bubble column reactor equipped with a glass gas frit at the bottom (shown in Fig.1) with available volume of 300 ml solution in a water bath, which can change reaction temperature. The top of the reactor had two openings providing connections to a pipette for feeding or sampling and venting the gas. All connections including tubing and valves were composed of Teflon or glass without metals. The pipes between U-tube and reactor were packed with resistance coils to provide temperature.

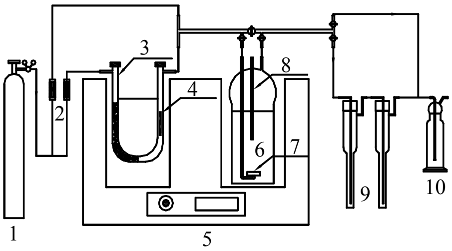

Fig.1 Schematic diagram of the experimental apparatus

1: N_2; 2: Rotameter; 3: U-tube; 4: Mercury permeation tube; 5: Water bath; 6: Reactor; 7: Gas diffuser; 8: Sample out; 9: Impinger; 10: Waste gas scrubber

Mercury concentration was obtained from a permeation tube inside a glass U-tube, immersed in one water bath (T_1) at a pure nitrogen flow of 120 ml/min. The total flow (G) was 1 L/min diluted by nitrogen. In each typical experiment, the temperature of water baths were adjusted to the constant values, and then 250 ml water or other background solution was put into the reactor immersed in the other water bath (T_2). Finally, Hg^0/N_2 mixed gas was passed over solution in the reactor and the vent gas was scrubbed by acidic $KMnO_4$. After about 2 h, the mixed gas was passed over 10% (v/v) H_2SO_4-4% (w/v) $KMnO_4$ solution adopted by Environmental Protection Agency (EPA), 10 ml each, inside two impingers in series for 1 min. Then the vent gas was switched to waste gas scrubber again. The process was repeated thrice at 10 min intervals. The average value was the initial Hg^0 concentration in the inlet. If these three values gave stable readings, known amounts of potassium persulfate solution were sequentially injected into the reactor using a syringe with a slim Teflon tube. The outlet mercury concentration was sampled in two impingers in series at intervals.

Analytical methods

One millilitre samples were withdrawn by glass syringes at different reaction time, filtered twice through 0.22 μm membrane filters after adding a drop of NaCl (1.0 mol/L) and then analyzed for sulfate using Metrohm 792 Basic Ion Chromatograph (IC) equipped with a Metrosep A Supp 4 column (250 mm×4.0 mm), a Metrosep A Supp 4/5 guard column, and a conductivity detector. The eluent (flow rate=1.0 ml/s) was a standard Metrohm mixture of 2.0 mmol/L sodium carbonate and 1.0 mmol/L sodium bicarbonate. pH value was measured with digital pH meter (JENCO, Shanghai, China). Liquid samples in two impingers were put into the test tubes. Before analysis, several drops of hydroxylamine hydrochloride were put into the test tubes until the solution color turned clear. Five millilitres sample was put into a mercury reactor, an accessory of QM201B mercury analyzer (Qingan Instrument Company, Suzhou, China), and reduced by 1 ml 10% (w/v) $SnCl_2$-20% (v/v) solution. Mercury vapor produced by reaction was flushed out to the absorption cell in the analyzer by air.

RESULTS AND DISCUSSION

Effect of initial pH value

Fig.2 shows the influence of initial pH value on the Hg^0 removal. While initial pH was 7, the removal efficiency of Hg^0 reached nearly 54.4% in 40 min reaction, followed by 30.8% for 1 mol/L HNO_3 solution. While NaOH concentration was 0.1 mol/L, Hg^0 removal efficiency was extremely low, and no more than 5.5%. It was obvious that the presence of both H^+ and OH^-, especially OH^-, greatly weaken the Hg^0 oxidation when silver nitrite was absent.

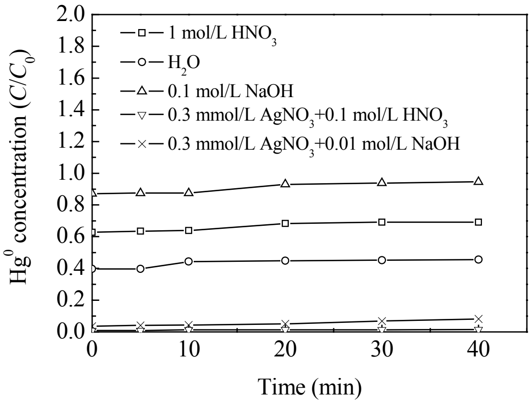

Fig.2 Effect of pH on Hg^0 removal
T_1=50 °C, C_{KPS}=10.0 mmol/L, T_2=25 °C, G=1 L/min

$S_2O_8^{2-}$ could be decomposed catalyzed by acid. The mechanism is given by the following reactions (House, 1962):

$$S_2O_8^{2-}+H^+ \rightarrow HS_2O_8^- \quad (1)$$

$$HS_2O_8^-+H_2O \rightarrow HOOSO_3^-+HSO_4^-+H^+ \quad (2)$$

$$HOOSO_3^-+H_2O \rightarrow HSO_4^-+H_2O_2 \quad (3)$$

$$2H_2O_2 \rightarrow 2H_2O+O_2 \quad (4)$$

$S_2O_8^{2-}$ decomposes unsymmetrically by reaction with H_2O because of the influence of H^+ and involved the breaking of the O-S bond. At the same time, $S_2O_8^{2-}$ can also react with H_2O in neutral solutions, which was considered the product of $SO_4^-\cdot$ is the rate limiting step (House, 1962). $S_2O_8^{2-}$ decomposes to produce $SO_4^-\cdot$ by breaking of the O-O bond.

$$S_2O_8^{2-}+H_2O \rightarrow HSO_4^-+SO_4^-\cdot+OH\cdot \quad (5)$$

$$SO_4^-\cdot+H_2O \rightarrow HSO_4^-+OH\cdot \quad (6)$$

$$2OH\cdot \rightarrow H_2O_2 \rightarrow H_2O+\frac{1}{2}O_2 \quad (7)$$

So free radicals can produce in neutral solutions. On the contrary, no or little available species are produced in acidic aqueous solutions. Dogliotti and Hayon (1967) reported that sulfate radicals dominated in the photolysis of persulfate ions in neutral and acidic aqueous solutions. However, in alkaline solutions, sulfate radicals are converted into hydroxyl radicals (OH·). It was shown that sulfate radicals (2.5~3.1 V) are stronger oxidants than hydroxyl radicals (1.985, 2.38 or 2.72 V in acidic solution), especially at elevated pH (Anipsitakis and Dionysiou, 2004). But the results in alkaline solutions, under which KPS would not decompose easily because of lack of available H_2O or H^+, indicated that no free radicals were produced without light. But the difference was almost eliminated by adding Ag^+, regardless of acid or alkaline solutions.

Effect of $K_2S_2O_8/AgNO_3$ concentration

The mercury removal profiles with different initial potassium persulfate concentration and other fixed parameters are shown in Fig.3.

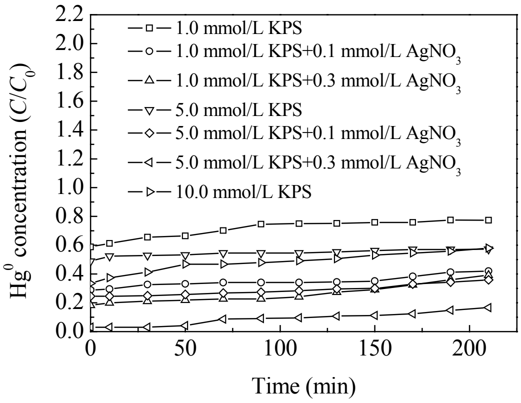

Fig.3 Effect of KPS/$AgNO_3$ concentration on Hg^0 removal
pH=7, T_1=50 °C, T_2=55 °C, G=1 L/min

Hg^0 concentration at the reactor outlet decreases sharply at the beginning, and then reaches a plateau value nearly as the reaction proceeded due to the excess of KPS. The highest reaction rate occurred at the highest initial concentration of potassium persulfate.

For instance, silver nitrate concentration was 0.3 mmol/L, after 210 min reaction, the removal rate of mercury reached 47.03% and 94.16% when the potassium persulfate concentrations were 1.0 mmol/L and 5.0 mmol/L, respectively. When silver nitrate concentration was 0.1 mmol/L, similar results were obtained. 1.0 mmol/L KPS was not enough to removal effectively, regardless of the concentration of silver nitrate.

As shown in Fig.3, silver nitrate accelerates dramatically the reaction rate and increases mercury removal efficiency due to the production of high reactive transition ions, Ag^{2+} and Ag^{3+} (see Eq.(8) and Eq.(9)). Furthermore, mercury removal efficiency increases with increasing silver nitrate concentration. Mercury removal is only 42.8% without silver nitrate, while mercury removal efficiency could reach 64.0% and 83.3% corresponding to 0.1 and 0.3 mmol/L $AgNO_3$ for 5.0 mmol/L KPS, respectively. 0.1 mmol/L $AgNO_3$ is not enough to remove mercury completely for 5.0 mmol/L KPS. The more the silver nitrate concentration is, the higher the mercury removal in our study. The reaction rate is proportional to the KPS concentration and to the $AgNO_3$ concentration (House, 1962).

Effect of TBA

Fig.4 gives the results on effect of TBA on mercury removal. Generally speaking, radical scavenger includes mainly CO_3^{2-} and HCO_3^{2-}, and tertiary butanol, which react with OH· at rates of 4.2×10^8 L/(mol·s), 1.5×10^7 L/(mol·s) and 5.0×10^8 L/(mol·s) (Langlais *et al.*, 1991) respectively. Among them, tertiary butanol is the most active. To ascertain whether OH· and $SO_4^- \cdot$ took participate in the reaction between Hg^0 and potassium persulfate, 50 mmol/L tertiary butanol was put into the solution. According to the figure, the removal efficiency decreased from 58.0% to 34.8% for 0.1 mmol/L $AgNO_3$, after 210 min reaction, which was attributed to the influence of tertiary butanol. Besides, the removal efficiency was comparatively low, after adding TBA, no matter the concentration of $AgNO_3$. Even when a large quantity of TBA was present, removal efficiency still reached 34.8% because KPS could attack Hg^0 directly due to the high redox potential. Competitive reaction between TBA and Hg^0 with KPS is the other reason. So it can be concluded that the role of free radicals could be of significance in the reaction.

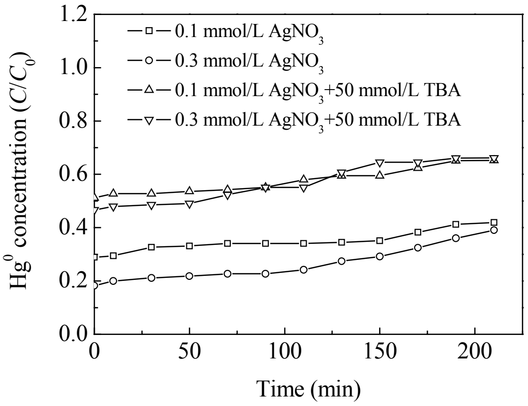

Fig.4 Effect of TBA on Hg^0 removal
pH=7, T_1=50 °C, C_{KPS}=1.0 mmol/L, T_2=55 °C, G=1 L/min

Effect of reaction temperature

Fig.5 of the effect of reaction temperature T_2 on Hg^0 removal indicates that removal efficiency decreases with increasing temperature. Lower temperature enhances mercury removal. The removal rate was reduced from 70.5%, 78.3% at 25 °C to 58.0%, 60.9% at 55 °C for 0.1 mmol/L and 0.3 mmol/L $AgNO_3$, respectively. The effect of reaction temperature exceeds that of $AgNO_3$ concentration for 1.0 mmol/L KPS. This is probably due to the low KPS concentration. The energy of O-O bond is 33.5 kJ/mol and the activation energy is (121±12) kJ/mol (Price and Clifton, 1996), which is in reasonable agreement with 130 kJ/mol in previous work (Kolthoff and Miller, 1951) when potassium persulfate decomposed to radical $(SO_4^- \cdot)$, whose potential was as high as 2.6 V (Kolthoff and Miller, 1951). Due to the high activation energy, reaction rate is quite slow at room temperature and acceptable rate can only be achieved over 50 °C. But the rate constant of $SO_4^- \cdot$ recombination is $(2.7\pm0.2)\times10^9$ L/(mol·s) (Ivanov *et al.*, 2000), which is the same order compared to OH· recombination (5.5×10^9 L/(mol·s)). But the efficiency of $KPS/AgNO_3$ is comparable with that of $KMnO_4/H_2SO_4$ (not shown here) as oxidants for Hg^0 removal. The latter reaction rate is in the range of 10^7 L/(mol·s) (Zhao and Rochelle, 1996). As a result, the effect of temperature was investigated from two inverse aspects. One is that high temperature can elevate reaction rate, but relatively low temperature, 55 °C, is not enough to overcome activation energy, the other increase the chance of free radical recombination. Furthermore, the net effect is probably governed by the latter.

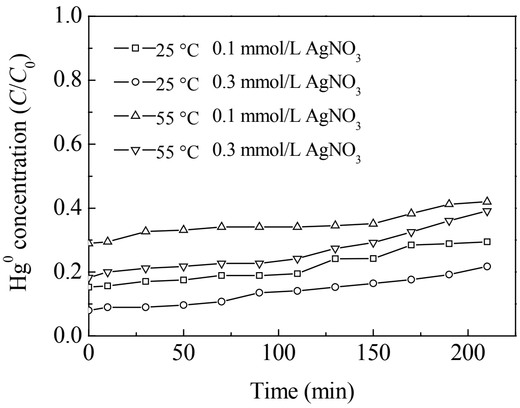

Fig.5 Effect of reaction temperature on Hg^0 removal
pH=7, T_1=50 °C, C_{KPS}=1.0 mmol/L, G=1 L/min

Effect of Hg^0 concentration in the inlet

Due to various Hg^0 concentrations in the flue gases, its effect should be considered. Different Hg^0 concentrations can be achieved by regulating temperature, T_1, of the water bath. The results are shown in Fig.6. It was evident that elevating Hg^0 concentration in gas phase increased the concentration gradient (gas phase force), which would speed up reaction rate and result in high removal efficiency. After 210 min reaction, the removal efficiency of Hg^0 reached 64.0% and 80.9% for 0.1 mmol/L $AgNO_3$ and 5.0 mmol/L KPS when T_1 was 50 °C and 70 °C respectively. The difference was reduced for 0.3 mmol/L $AgNO_3$. The efficiency was 83.3% and 90.9% corresponding to 50 °C and 70 °C.

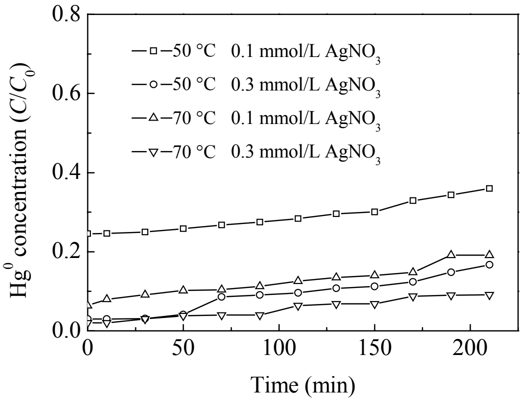

Fig.6 Effect of Hg^0 concentration in the inlet on Hg^0 removal
pH=7, T_2=55 °C, C_{KPS}=5.0 mmol/L, G=1 L/min

Reaction mechanism

It was postulated that persulfate anion ($S_2O_8^{2-}$) can be thermally or chemically catalyzed by transition metal ions to produce a powerful oxidant known as the sulfate free radical ($SO_4^{-}\cdot$) (House, 1962), which can extract a hydrogen from water to give hydroxyl radicals (OH·) and potentially destroy organic contaminants within the soil mass by in situ chemical oxidation.

The roles of free radicals and transition species in the reaction were investigated by adding 50 mmol/L TBA discussed above, which proved the postulation. Fig.7 indicates that the production of SO_4^{2-} almost linearly increases, while pH decreases with time.

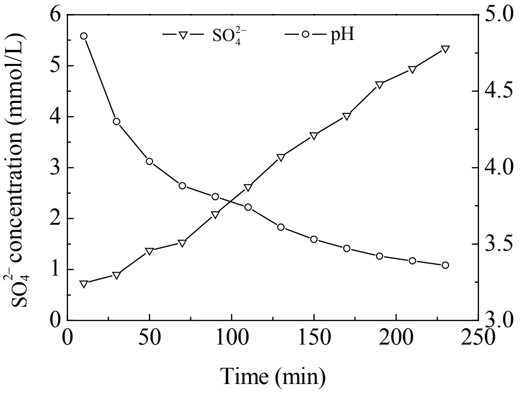

Fig.7 sulfate anion concentration and pH figure as time increases
pH=7, T_1=50 °C, T_2=55 °C, C_{KPS}=5.0 mmol/L, C_{CN}=0.3 mmol/L, G=1 L/min

The reactions of persulfate ions with various organic and inorganic compounds have been extensively studied (Kolthoff and Miller, 1951; House, 1962; Berlin, 1986). Persulfate oxidation is generally conducted under heat-, photo- or metal-catalyzed conditions because the oxidation rates can be greatly accelerated. Highly reactive species such as sulfate radicals ($SO_4^{-}\cdot$) and hydroxyl radicals (OH·) are generated as a result of photolysis or heat decomposition of persulfate ions in aqueous phases (Nosov, 1966;

Dogliotti and Hayon, 1967; Hayon and McGarvey, 1967; Berlin, 1986; Tanner and Osman, 1987).

However, persulfate oxidation at atmospheric temperature is usually not effective. Persulfate is commonly used with UV light, catalyzed by transition metals or under high temperature in order to initiate or enhance its radical oxidation mechanisms (as Eq.(8)~ Eq.(16) show). Sulfate radicals, formed from heat or chemically catalyzed by silver ion decomposition of persulfate (Eq.(8)), may initiate a series of radical-transfer chain reactions (Eq.(9)~ Eq.(12)) (Berlin, 1986), where elemental mercury (Eq.(13) and Eq.(14)) is mainly removed. At the same time, instable transition species (Ag^{3+} and Ag^{2+}) probably also contribute to mercury removal via OH· formation. Chain termination occurs in some situations to result in radical recombination (Eq.(15) and Eq.(16)).

$$Ag^{+}+S_2O_8^{2-} \rightarrow SO_4^{-}\cdot+SO_4^{2-}+Ag^{2+} \tag{8}$$

$$Ag^{+}+S_2O_8^{2-} \rightarrow 2SO_4^{2-}+Ag^{3+} \tag{9}$$

$$Ag^{+}+Ag^{3+} \rightarrow 2Ag^{2+} \tag{10}$$

$$SO_4^{-}\cdot+H_2O \rightarrow HSO_4^{-}+OH\cdot \tag{11}$$

$$Ag^{2+}+H_2O \rightarrow OH\cdot+Ag^{+}+H^{+} \tag{12}$$

$$2SO_4^{-}\cdot+Hg \rightarrow Hg^{2+}+2SO_4^{2-} \tag{13}$$

$$2OH\cdot+Hg+2H^{+} \rightarrow Hg^{2+}+2H_2O \tag{14}$$

$$2OH\cdot \rightarrow H_2O_2 \rightarrow H_2O+\frac{1}{2}O_2 \tag{15}$$

$$2SO_4^{-}\cdot \rightarrow S_2O_8^{2-} \tag{16}$$

The most probably stoichiometry between Hg^0 and KPS is 1:1, as shown below:

$$Hg+K_2S_2O_8=HgSO_4+K_2SO_4 \tag{17}$$

After 210 min reaction was completed, it was just only 1.75×10^{-4} mmol/L KPS concentration that could reach the theoretical value. Obviously, 5.0 mmol/L KPS was of excess. Fig.7 shows that 2.67 mmol/L KPS was consumed, which accounted for 53.4%. These results may be explained by the facts that free radicals would recombine or react with other reagents instead of Hg^0, and that, as a result, chain reaction ceased. The other reason is that KPS catalyzed by Ag^+ decomposition continued to proceed without any pretreatment before IC analysis.

CONCLUSION

Potassium persulfate does not remove Hg^0 effectively, no more than 60% without Ag^+ after 210 min (Fig.3). Nevertheless, Ag^+ catalyzed KPS decomposition could greatly increase Hg^0 removal. Influence of pH without $AgNO_3$ was remarkable; however, mercury removal was hardly affected as $AgNO_3$ was present, regardless of pH. Removal efficiency increases with increasing concentration of potassium persulfate and silver nitrate. Relatively low temperature is favorable for removal efficiency in our study, while radical scavenger inhibits Hg^0 absorption. High Hg^0 concentration also promotes removal. According to the product of oxidation, such as SO_4^{2-} and H^+, the possible free radical reaction mechanism of gaseous mercury absorption in potassium persulfate catalyzed by silver nitrate was also presumed. Direct reaction combined with indirect radical oxidation promoted mercury removal.

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