

Highly efficient oxidation of chromium (III) with hydrogen peroxide in alkaline medium

Hao Peng, Jing Guo, Gang Li, Qinzhe Cheng, Yuju Zhou, Zuohua Liu and Changyuan Tao

ABSTRACT

Many technologies have been proposed to oxidize chromium, such as roasting-water leaching technology and hydrometallurgical methods such as pressure oxidative leaching coupled with oxygen, ozone, permanganate and ferrate, but the problems associated with the high temperature, low overall resource utilization efficiency, high energy consumption, and the environmental pollution, still remain unsolved. This paper focuses on the oxidation process of chromium (III) with hydrogen peroxide (H₂O₂) in an alkaline medium. The effect of parameters including dosage of H₂O₂, dosage of NaOH, reaction time, reaction temperature and stirring rate on the oxidation efficiency of chromium were investigated. The oxidation efficiency was significantly affected by the dosage of H₂O₂ and NaOH, reaction time and reaction temperature took second place; last was the stirring rate. Oxidation efficiency was nearly 100% under the optimal conditions: volume ratio of H₂O₂ to mass of Cr₂(SO₄)₃ of 2.4 mL/g, mass ratio of NaOH to Cr₂(SO₄)₃ 0.6 g/g, reaction time of 90 min, reaction temperature of 90 °C and stirring rate of 500 rpm.

Key words | advanced oxidation, alkaline medium, chromium, H₂O₂

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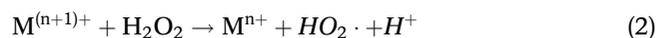
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INTRODUCTION

Chromium is an important strategic resource widely used in chemical manufacturing, electroplating, leather tanning, metal corrosion inhibition, and pigment production (Gupta *et al.* 2011; Jin *et al.* 2013; Steffy *et al.* 2013; Wessel & Dronskowski 2013; Saleh & Al-Absi 2017; Saleh *et al.* 2017a; Saleh *et al.* 2017b; Xin *et al.* 2017; Yufen *et al.* 2017; Peng *et al.* 2018a; Peng *et al.* 2018b; Saleh 2018; Saleh *et al.* 2018; Abubakar *et al.* 2019). Chromium mainly exists as chromium (III) (Cr (III)) and chromium (VI) (Cr (VI)) which are stable and show different characteristics (Gupta *et al.* 2011; Saleh *et al.* 2016; Ali *et al.* 2017; Adio *et al.* 2019). Chromium (VI) is emitted by some industrial activities like petroleum refining, battery production and electroplating, etc. (Xu *et al.* 2004; Peng *et al.* 2015; Peng *et al.* 2016; Qing *et al.* 2017). And it mainly exists as dichromate (Cr₂O₇²⁻) in acidic solution and chromate (HCrO₄⁻, CrO₄²⁻) in alkaline solution.

Many hydrometallurgical processes have been proposed to recover chromium (Shiyuan *et al.* 2016; Yuan *et al.* 2016; Hongrui *et al.* 2017; Qing *et al.* 2017; Spanka *et al.* 2018). Conventionally, sodium roasting-water leaching technology, calcium roasting-acid leaching (Xue *et al.* 2017b), and

hydrometallurgical methods such as pressure oxidative leaching coupled with oxygen, ozone, permanganate and ferrate have been developed (Zhang *et al.* 2014; Li *et al.* 2015; Kim *et al.* 2016; Jing *et al.* 2017; Xue *et al.* 2017a), but the problems associated with the high temperature salt roasting technologies, including the low overall resource utilization efficiency, the high energy consumption, and the environmental pollution (toxic gas, and waste water), still remain unsolved. Hydrogen peroxide (H₂O₂) is widely used in the treatment of wastewater as an oxidant like the Fenton-like method (Li *et al.* 2018; Wang & Liu 2018; Xu *et al.* 2018; Zhao *et al.* 2018). H₂O₂ reacts with transition metal ions to generate ·OH according to Equations (1) and (2), which can be treated as the mechanism of Fenton-like reaction (Rahim Pouran *et al.* 2016; Wang *et al.* 2016).



$\cdot\text{OH}$ is a powerful oxidizing reagent with high oxidation potential and it exhibits a faster rate of oxidation reaction (Gogate & Pandit 2004). $\cdot\text{OH}$ attacks the organic pollutant molecules, leading to their degradation.

In this paper, H_2O_2 acted as the oxidant with the direct advanced oxidation process applied to oxidize chromium (III) to chromium (VI). The effect of dosage of H_2O_2 , dosage of NaOH , reaction time, reaction temperature and stirring rate on the oxidation efficiency of chromium were preferentially examined.

EXPERIMENTAL

Materials

Chromium sulfate ($\text{Cr}_2(\text{SO}_4)_3$), hydrogen peroxide (H_2O_2), and sodium hydroxide (NaOH) of analytical grade were purchased from Kelong Co., Ltd, Chengdu, China, and used as received without purification. Deionized water used in the experiments was produced by a water purification system (HMC-WS10).

Apparatus and procedures

All experiments were performed in a glass beaker with a thermostatic mixing water bath pot. A predetermined amount of chromium sulfate and deionized water was added to the beaker to produce a homogeneous solution under constant stirring. The solution was heated to a predetermined temperature. Next, the sodium hydroxide was added to the reactor. Finally, H_2O_2 was added to the medium manually. After the required reaction time, the concentration of chromium (III) and chromium (VI) was determined by inductively coupled plasma-optical emission spectrometry (ICP-OES, PerkinElmer Optima 6300DV). The oxidation efficiency (η) of chromium was calculated as Equation (4):

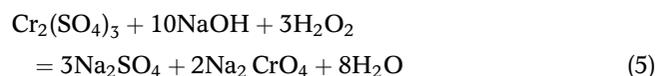
$$\eta = \frac{C_1 \cdot V_1 - C_2 \cdot V_2}{C_1 \cdot V_1} \times 100\% \quad (4)$$

where, C_1 and C_2 are the concentration of chromium in the solution before and after the experiment; V_1 and V_2 are the volume before and after the experiment, mL.

RESULTS AND DISCUSSION

Technology principle

The main reaction during the oxidation process was between chromium sulfate, hydrogen peroxide and sodium hydroxide, which reacted as Equation (5) (Dalun & Jianhua 2002). The ΔG_T^θ of Equation (5) at different temperatures could be calculated with $\Delta_f H_{298}^\theta$, S_{298}^θ and C_p at 298 K, shown in Equations (6)–(8) (Dalun & Jianhua 2002).



$$\Delta G_T^\theta = \Delta H_T^\theta - T\Delta S_T^\theta \quad (6)$$

$$\Delta H_T^\theta = \Delta H_{298}^\theta + \int_{298}^T \Delta C_p dT \quad (7)$$

$$\Delta S_T^\theta = \Delta S_{298}^\theta + \int_{298}^T \frac{\Delta C_p}{T} dT \quad (8)$$

Equation (9) was obtained by merging Equations (6)–(8).

$$\Delta G_T^\theta = \Delta H_{298}^\theta - T\Delta S_{298}^\theta + \int_{298}^T \Delta C_p dT - T \int_{298}^T \frac{\Delta C_p}{T} dT \quad (9)$$

The specific heat capacity, C_p , was calculated as Equation (10)

$$C_p = a + b \times 10^{-3}T + c \times 10^5 T^{-2} + d \times 10^{-6}T^2 \quad (10)$$

And also, ΔC_p was calculated as Equation (11).

$$\Delta C_p = \Delta a + \Delta b \times 10^{-3}T + \Delta c \times 10^5 T^{-2} + \Delta d \times 10^{-6}T^2 \quad (11)$$

Then ΔG_T^θ was calculated using Equation (12).

$$\begin{aligned} \Delta G_T^\theta = \Delta H_{298}^\theta - T\Delta S_{298}^\theta - \\ T \int_{298}^T \frac{dT}{T^2} \int_{298}^T (\Delta a + \Delta b \times 10^{-3}T + \Delta c \times 10^5 T^{-2} + \Delta d \times 10^{-6}T^2) dT \end{aligned} \quad (12)$$

Integrate:

$$\begin{aligned} \Delta G_T^\theta = \Delta H_{298}^\theta - T\Delta S_{298}^\theta - T \left\{ \Delta a \left(\ln \frac{T}{298} + \frac{298}{T} - 1 \right) \right. \\ \left. + \Delta b \times 10^{-3} \left[\frac{1}{2T} (T - 298)^2 \right] + \frac{\Delta c \times 10^5}{2} \left(\frac{1}{298} - \frac{1}{T} \right)^2 \right. \\ \left. + \Delta d \times 10^{-6} \left(\frac{T^2}{6} + \frac{298^3}{3T} - \frac{298^2}{2} \right) \right\} \end{aligned} \quad (13)$$

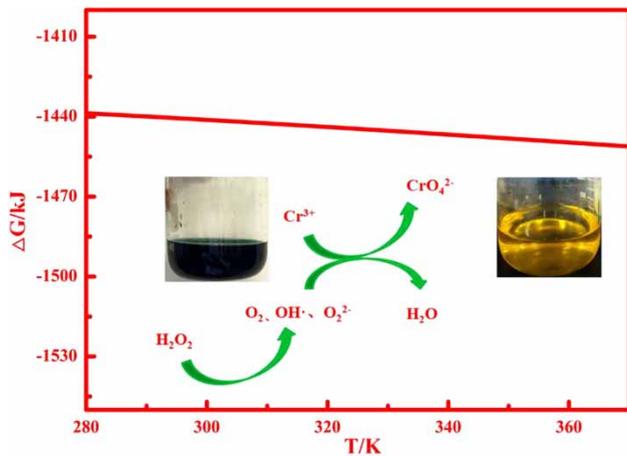


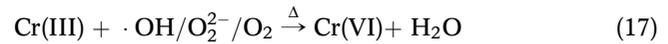
Figure 1 | Relationship between ΔG and temperature of oxidation of chromium (III).

The $\Delta_f H_{298}^\theta$, ΔS_T^θ , a, b, c and d in Equation (13) could be obtained from the reference (Dalun & Jianhua 2002). The calculation result is shown in Figure 1.

The results shown in Figure 1 indicate that the oxidation of Cr^{3+} with hydrogen peroxide is feasible in thermodynamics as the ΔG was negative (Xiancai et al. 2005).

During the oxidation process, $\cdot\text{OH}$, $\text{O}_2^{\cdot-}$ and O_2 were produced by heating the H_2O_2 solution. These were

responsible for the oxidation of chromium (III) according to Equations (14)–(17).



Effect of volume ratio of H_2O_2 to mass of $\text{Cr}_2(\text{SO}_4)_3$

The effect of volume ratio of H_2O_2 to mass of $\text{Cr}_2(\text{SO}_4)_3$ (VR) on the oxidation efficiency of chromium was preferentially examined under the following conditions: reaction temperature of 90 °C, reaction time of 90 min, mass ratio (MR) of NaOH to $\text{Cr}_2(\text{SO}_4)_3$ of 0.6 g/g and stirring rate of 500 rpm. The oxidation efficiency and the concentration of Cr (III) and Cr (VI) in the solution are summarized in Figure 2.

The results in Figure 2 show that the Cr^{3+} could be oxidized to CrO_4^{2-} effectively in an alkaline medium and the

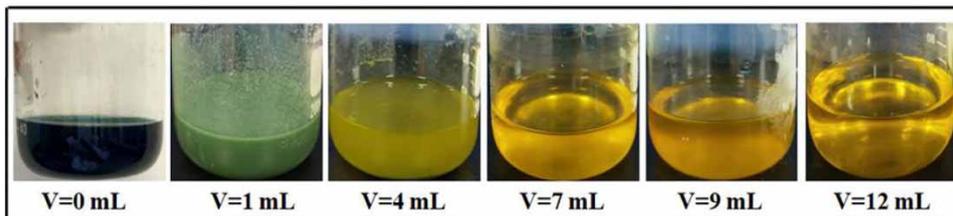
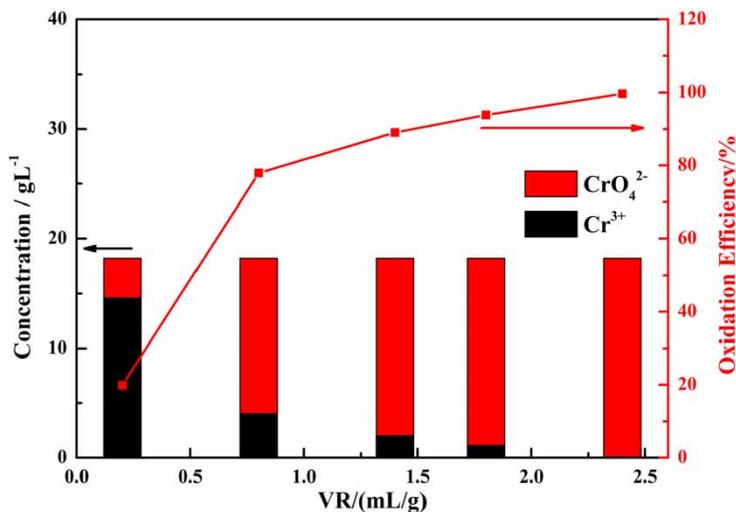


Figure 2 | Effect of VR on oxidation efficiency of chromium (reaction temperature 90 °C, reaction time of 90 min, mass ratio of NaOH to $\text{Cr}_2(\text{SO}_4)_3$ of 0.6 g/g, stirring rate of 500 rpm, VR was set as 0.2 ($V(\text{H}_2\text{O}_2) = 1 \text{ mL}$), 0.8 ($V(\text{H}_2\text{O}_2) = 4 \text{ mL}$), 1.4 ($V(\text{H}_2\text{O}_2) = 7 \text{ mL}$), 1.8 ($V(\text{H}_2\text{O}_2) = 9 \text{ mL}$) and 2.4 ($V(\text{H}_2\text{O}_2) = 12 \text{ mL}$)).

dosage of H_2O_2 had a significant effect on the oxidation of Cr^{3+} . The oxidation efficiency was linearly increasing with the increase of VR. The chromium in the solution was Cr^{3+} in the original solution and the solution was dark green in color. The color of the solution and formation of chromium changed with the addition of NaOH and H_2O_2 . When VR = 0.2 mL/g, chromium mainly existed as $\text{Cr}(\text{OH})_3$ in the alkaline medium and the color of the solution was green. As the VR increased, the reaction occurred in the solution according to Equation (14) and Cr^{3+} was oxidized to CrO_4^{2-} ; the solution was changed to yellow. When the VR = 2.4 mL/g, the chromium in the solution almost all existed as CrO_4^{2-} and the oxidation efficiency was nearly 100%.

Effect of mass ratio of NaOH to $\text{Cr}_2(\text{SO}_4)_3$

The Cr^{3+} in the acid medium was hard to oxidize except if the oxidant was persulfate salt. While it was easy for it to be oxidized in an alkaline medium (Banwen & Yuji 1998), the dosage of NaOH had a big influence on the oxidation efficiency of Cr^{3+} . The effect of dosage of NaOH on the oxidation efficiency and the concentration of Cr (III) and

Cr (VI) in the solution was preferentially studied under the following conditions: VR of 2.4 mL/g, reaction temperature of 90 °C, reaction time of 90 min and stirring rate of 500 rpm. During the oxidation process, NaOH was not only an important reactant but also it provided an alkaline medium. The results shown in Figure 3 indicate that the dosage of NaOH significantly affected the oxidation process. The oxidation efficiency was only 36.92% at MR = 0.2 and then increased to 99.67% as MR increased to 0.6.

Effect of reaction temperature

The reaction temperature (T) was another important parameter that had a significant influence on the oxidation process of chromium, both thermodynamic and kinetic. Figure 4 summarizes the effect of reaction temperature on the percentage of Cr (III) and Cr (VI) in a reactant solution under the standard conditions: VR of 2.4 mL/g, reaction time of 90 min, MR of 0.6 g/g and stirring rate of 500 rpm.

It could be concluded that reaction temperature was an important parameter during the oxidation process according to the reaction equations, and was consistent with the

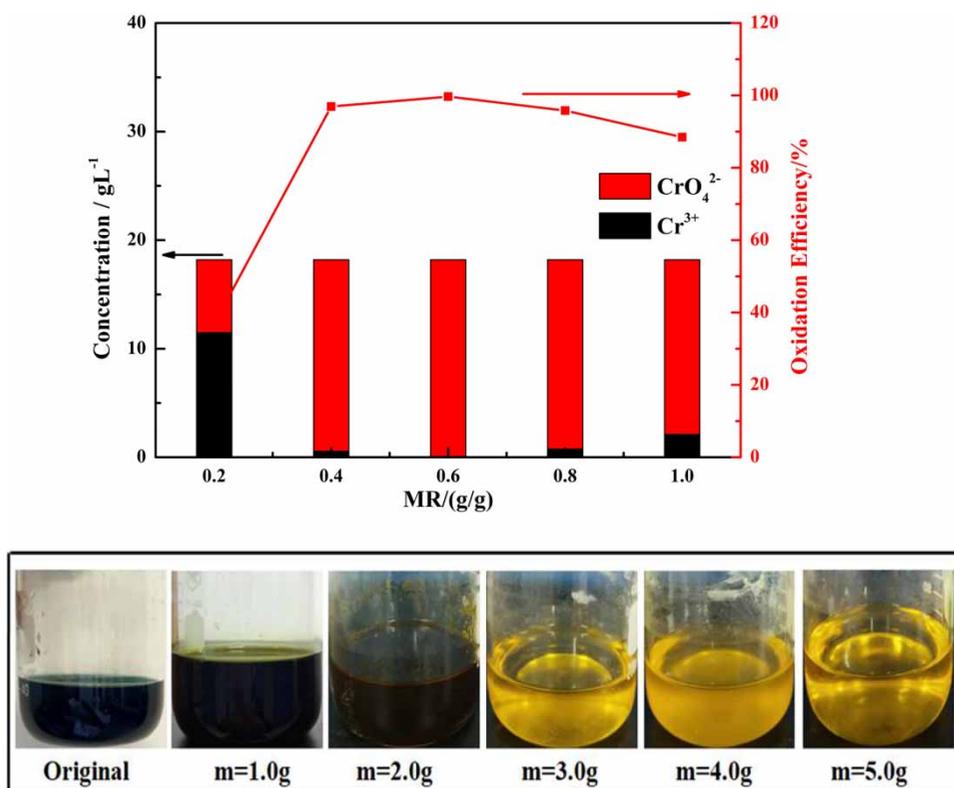


Figure 3 | Effect of dosage of NaOH on oxidation efficiency of chromium (VR of 2.4 mL/g, reaction temperature of 90 °C, reaction time of 90 min and stirring rate of 500 rpm; MR was set as 0.2 (m(NaOH) = 1.0 g), 0.4 (m(NaOH) = 2.0 g), 0.6 (m(NaOH) = 3.0 g), 0.8 (m(NaOH) = 4.0 g) and 1.0 (m(NaOH) = 5.0 g)).

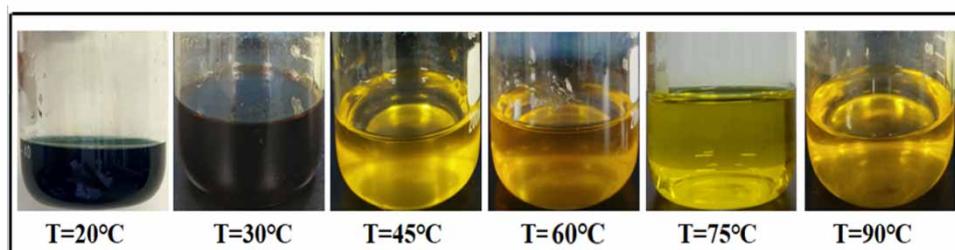
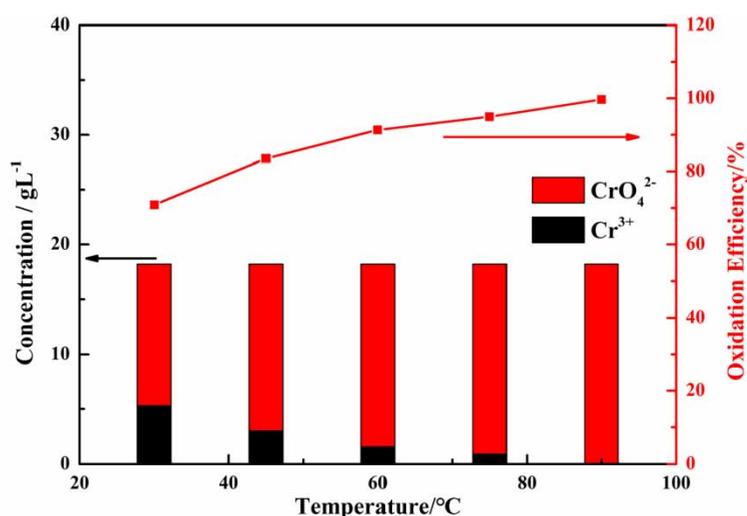


Figure 4 | Effect of reaction temperature on oxidation efficiency of chromium (VR of 2.4 mL/g, reaction time of 90 min, MR of 0.6 g/g and stirring rate of 500 rpm).

results shown in Figure 4. The oxidation efficiency was about 70% at 30 °C and then increased linearly with the increase of reaction temperature. The results were consistent with the results shown in Figure 1, in which the oxidation of Cr³⁺ happened easily at low temperature. In a high temperature condition, reactants like Cr³⁺, ·OH, O₂²⁻ and O₂ had high reactivity and the reaction happened more easily. Therefore, high reaction temperature was an essential condition during the oxidation process.

Effect of reaction time

The effect of reaction time on the oxidation efficiency and the concentration of Cr (III) and Cr (VI) in the solution were preferentially examined under the following conditions: VR of 2.4 mL/g, stirring rate of 500 rpm, reaction temperature of 90 °C, and MR 0.6 g/g. The oxidation efficiency and the concentration of Cr (III) and Cr (VI) in the solution are summarized in Figure 5. It could be concluded that the reaction time has little influence on the oxidation efficiency of Cr³⁺, as the oxidation efficiency was about 80% at 30 min according to the results showed in Figure 5. Extending the reaction time could accelerate the oxidation

reaction and improve the oxidation efficiency as it was nearly 100% at 90 min.

Effect of stirring rate

The mixing performance was significantly affected by the stirring rate and stirring blade in the chemical process (Liu et al. 2014). The effect of stirring rate on the oxidation efficiency and the concentration of Cr (III) and Cr (VI) in the solution were preferentially examined under the following conditions: VR of 2.4 mL/g, reaction temperature of 90 °C, and MR 0.6 g/g and reaction time of 90 min. The results shown in Figure 6 suggest that stirring rate had little effect on the reaction. In other words, the oxidation reaction between Cr³⁺ and H₂O₂ in an alkaline medium was easily reacted and Cr³⁺ was easily oxidized to CrO₄²⁻ by H₂O₂.

Oxidation kinetics

The oxidation kinetic of chromium by H₂O₂ in an alkaline medium could be expressed as Equation (18):

$$-\frac{dC_{Cr}}{dt} = kC_{Cr}P_c \quad (18)$$

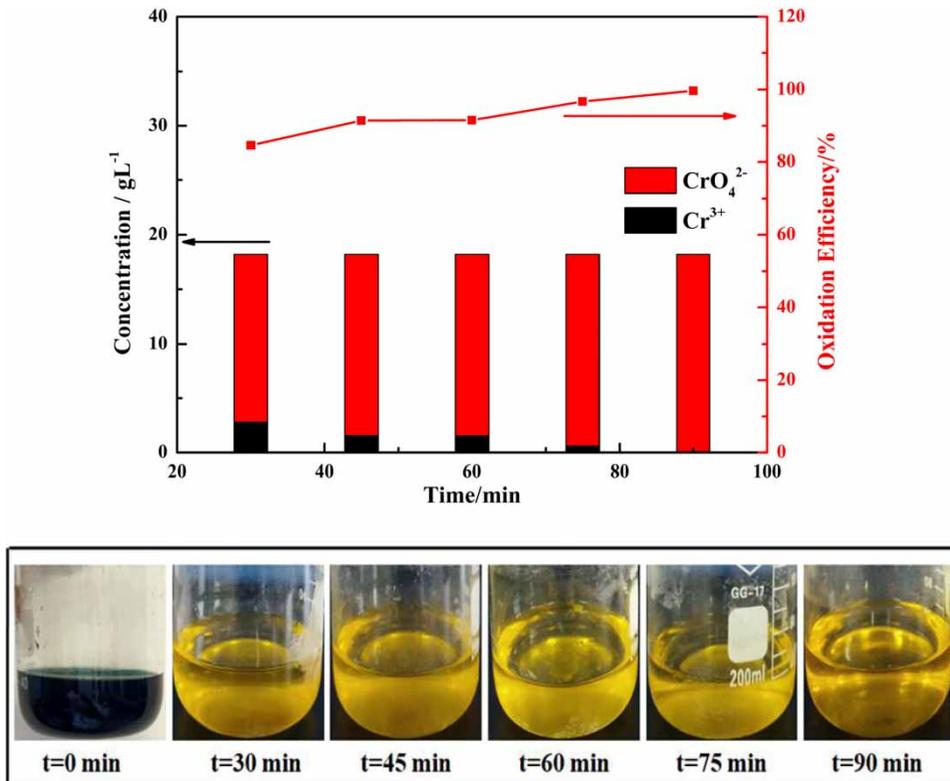


Figure 5 | Effect of reaction time on oxidation efficiency of chromium (VR of 2.4 mL/g, reaction temperature of 90 °C, stirring rate of 500 rpm, and MR 0.6 g/g).

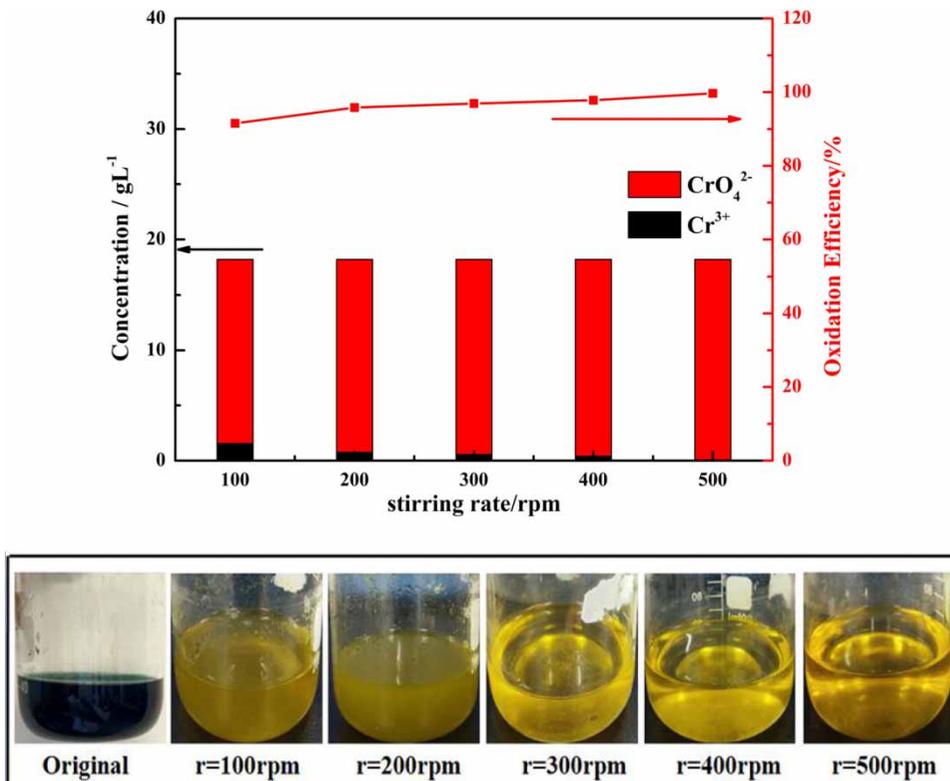


Figure 6 | Effect of stirring rate on oxidation efficiency of chromium (VR of 2.4 mL/g, reaction temperature of 90 °C, MR 0.6 g/g and reaction time of 90 min).

where, C_{Cr} is the concentration of Cr(VI); k is the reaction constant; P_c is the concentration of other reagents except Cr(VI), which was a constant as the concentration of other reagents were much larger.

Equation (18) was simplified as:

$$-\frac{dC_{Cr}}{dt} = k' C_{Cr} \quad (19)$$

Integrate:

$$\int_{C_{Cr0}}^{C_{Cr}} -\frac{dC_{Cr}}{C_{Cr}} = k' \int_0^t dt \quad (20)$$

$$\ln \frac{C_{Cr}}{C_{Cr0}} = \ln \eta = -k't \quad (21)$$

The oxidation efficiency of chromium at different temperatures is fitted in Figure 7, and the value of K was obtained, where K is the reaction rate constant corresponding to the slopes of the straight lines. Then the specific apparent activation energy could be calculated based on the Arrhenius equations (Equation (22)), the result are shown in Figure 8.

$$\ln K = \ln A - E_a/RT \quad (22)$$

where E_a is the apparent activation energy, A is the pre-exponential factor, and R is the molar gas constant.

The apparent activation energy of chromium oxidation is calculated to be 16.29 kJ/mol, the value was small and that indicated that chromium was easily oxidized by H_2O_2 in an alkaline medium.

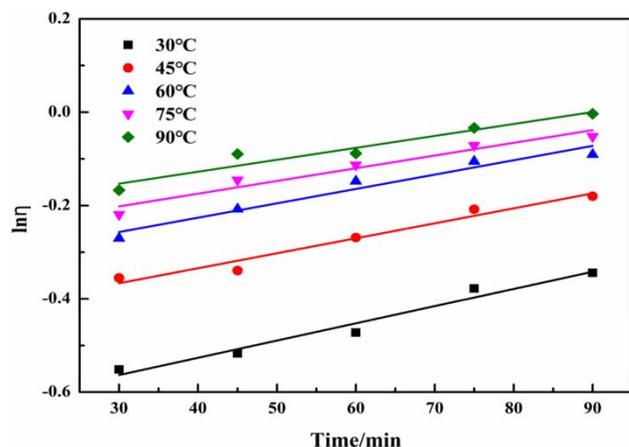


Figure 7 | Plot of oxidation kinetics of chromium at various reaction temperatures.

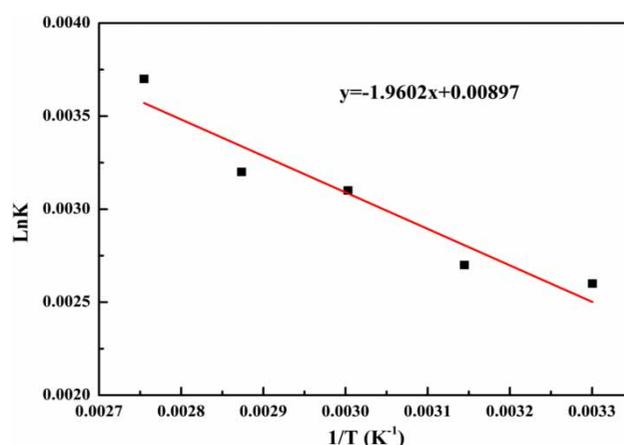


Figure 8 | Natural logarithm of oxidation constant versus reciprocal temperature of chromium.

CONCLUSIONS

In this paper, the oxidation process of Cr^{3+} with H_2O_2 in an alkaline medium was investigated. The results can be summarized as follows:

- (1) Chromium in low valence (Cr^{3+}) is easily oxidized to high valence (CrO_4^{2-}) with H_2O_2 in an alkaline medium. It is an environmentally-friendly technology showing substantial advantages in terms of energy efficiency, overall resource utilization efficiency and environmental pollution.
- (2) The dosage of H_2O_2 and NaOH had significant influence on the oxidation efficiency of chromium; reaction time and reaction temperature took second place; and last was the stirring rate.
- (3) The oxidation efficiency was nearly 100% under the optimal conditions: volume ratio of H_2O_2 to mass of $Cr_2(SO_4)_3$ of 2.4 mL/g, mass ratio of NaOH to $Cr_2(SO_4)_3$ 0.6 g/g, reaction time of 90 min, reaction temperature of 90 °C and stirring rate of 500 rpm.

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NOTES

The authors declare no conflicts of interest.

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