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Effect of forming technology on oxygen supply performance of oxygen candles in refuge spaces

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ABSTRACT
The present study investigated the forming technology of oxygen candles in a refuge space, for a more stable and reliable oxygen supply. Chlorine was found in the wet pressing process, revealing that dry pressing has a better forming effect on oxygen candles. Three types of oxygen candle blocks (OCBs) were pressed through dry pressing at pressure-application speeds of 0.1, 0.3, and 0.5 kN/s, respectively, for a pressing time of 10 min. From compression strength and combustion experiments, the ultimate compressive strengths (UCS) obtained for the three OCBs were 3.83, 5.48, and 5.83 kN. The stability coefficients of oxygen production (SCOP) were $U_1 = 0.966$, $U_2 = 0.724$, and $U_3 = 0.770$. From the results of these experiments and observation of combustion residue of OCBs, it was found that the stability of oxygen production decreases when the pressure-application speed within an appropriate pressure range is increased.
KEYWORDS: Oxygen candle; forming technology; dry pressing; pressure-applying speed;sStability of oxygen production

1. INTRODUCTION

In China, the current mode of oxygen supply to refuge spaces is through conventional forced air systems or compressed oxygen cylinders (Gao et al., 2012). However, in forced air systems oxygen is supplied through underground pipe networks, which usually have poor independence, as they might be destroyed in big mine accidents. Furthermore, compressed oxygen cylinders require regular maintenance, and are prone to quick discharge or explosions when exposed to high temperatures or fire (Jin et al., 2015). With technological developments regarding refuge spaces in China, the use of oxygen candles as an emergency oxygen source has attracted the attention of many scholars.

Oxygen candles are a type of oxygen supply equipment that releases oxygen through chemical reactions using chlorate or perchlorates as the source material. They have several advantages such as ease of use, large oxygen storage capacity (OSC), and fast oxygen production (Wang et al., 2010). The working principle of oxygen candles is the release of oxygen through thermal decomposition of chlorates (Fan et al., 2006). The decomposition reaction of chlorate is shown in equation (1):

$$2MClO_3 \xrightarrow{\Delta} 2MCl + 3O_2 \qquad (1)$$

where M is a type of alkali metal.

The heat required for the decomposition of chlorate is provided by the combustion of metal powders, such as iron powder and magnesium powder. The reaction for the combustion of magnesium is shown in equation (2):

$$2Mg + O_2 \longrightarrow 2MgO - Q \qquad (2)$$

At high temperatures or in the presence of impurities, a small amount of chlorate decomposes and releases chlorine, as shown in equation (3):

$$2MClO_3 \xrightarrow{\Delta/H_2O} M_2O + Cl_2 + \frac{5}{2}O_2 \qquad (3)$$

After passing through a filter material, the oxygen is directly released into the refuge space. Wang et al (2010) studied chlorine suppressants and chlorine gas filtering materials for oxygen candles. Zhang et al. (2013) studied the adsorption properties of carbon monoxide in emergency refuge facilities. Furthermore, Gao et al. (2015) studied an oxygen supply system and purification in a mine refuge space.

However, considering their use in refuge spaces, oxygen candles also present some shortcomings, such as heterogeneous, fast and uncontrollable oxygen release rate, and concentrated and high heat output from the reaction. In foreign countries, oxygen candles were extensively studied during the 1990s, followed by a systematic study of the catalysts, metal fuels, combustion fluctuations in oxygen candles, and the effect of particle size on combustion performance. In China, (Jin et al., 2014; Jin et al., 2015; Wang et al., 2014) the characteristics of oxygen supply in refuge spaces and the effect of M (M = catalysts, formula, candle structure) on oxygen

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supply performance (OSP) of oxygen candles were studied through thermogravimetric analyses and orthogonal experiments (Zhang et al., 2013). However, effective research is still lacking regarding the effect of forming technology on the OSP of oxygen candles.

In this study, the influence of different forming technologies on the OSP of oxygen candles was investigated by changing the forming technology, pressure-application speed, and holding time. The results have important significance in improving the stability and reliability of oxygen candles regarding OSP.

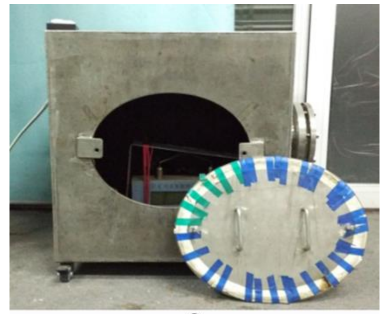
2. EXPERIMENT

2.1 Experimental Apparatus

A ball mill (KE-2L Planetary ball mill, Qidong HONGHONG Instrument Equipment Factory, China) was used to crush the components. Three oxygen candle blocks (OCBs) were prepared using a stainless steel die, which was designed and fabricated in-house (dimensions: 160 mm long，60 mm ID，80 mm OD), and an oil press (YES-300, Jinan Huaxing Testing Machine Co., Ltd., China).

After wet pressing, the OCBs were dried in an incubator (WM-BD temperature incubator, Shanghai Weiming Electronic Mechanical Equipment Co., Ltd., China).

The OCBs were subjected to reactions in a sealed chamber, which was also developed in-house (600 × 600 × 600 mm), as shown in Figure 1a. Oxygen concentration in the sealed chamber was monitored in real time using a mining multi-parameter measuring device (CD7, Beijing Zhong ShengZhou Mine Technology Center), which is shown in Figure 1b, and the dates were recorded using the KJ70N system in a computer.



a

b

Figure 1: Sealed chamber and CD7 mining multi-parameter measuring device.

Details of the chemicals used in the experiments are provided in Table 1.

Table 1: Chemicals used in the experiments.

Chemicals	Mol.wt.	Grades of purity (China)	Manufacturer
Sodium chlorate ($NaClO_3$)	106.44	EP	Sinopharm Chemical Reagent Co., Ltd.
Manganese (Mn)	54.94	2N	Johnson Malthey Chemicals Ltd.
Cobalt(III) oxide (Co_2O_3)	165.86	2N	Shanghai Fengshun Fine Chemicals Co., Ltd.
Kaolin	258.16	CP	Sinopharm Chemical Reagent Co., Ltd

2.2 Methods

Dry pressing and wet pressing experiments: The components (86.22% $NaClO_3$, 5.88% Mn, 4.90% Co_2O_3, 3% kaolin) (Jin et al., 2015) were processed in the ball mill for 5 min (particle size ≤ 100 μm), and a powder was obtained. In the dry pressing experiment, 145 g of the powder was placed on the stainless steel die, and subjected to a pressure of 35 kN at a pressure-application speed of 0.1 kN/s, and then the pressure was held for 5 min. They were then unloaded, and dried at 60°C for 12 h. Finally, the blocks were observed and their physical parameters were measured. In the wet pressing experiment, 5 ml and 10 ml of water were added to two groups of the powder weighing 145 g each and stirred homogeneously. The following processes were the same as in dry pressing.

Pressure-application speed and holding time experiments: in this study, three pressure speeds were defined, including slow pressing (0.1 kN/s), medium pressing (0.3 kN/s) and rapid pressing (0.5 kN/s). Three groups of the powder each weighing 145 g were subjected to slow pressing, medium pressing, and rapid pressing, and after reaching the target pressure (35 kN), it was maintained for 10 min (Table 3). They were unloaded and their physical properties were measured. Later, the combustion experiment was conducted in the sealed chamber, in which oxygen concentration was monitored in real time using the CD7. The experiment was stopped until the oxygen concentration no longer increased, and then the combustion residue of OCBs were observed. Finally, the OSP of all OCBs was determined.

2.3 Data processing

Processing of data from the pressure-application speed and holding time experiment: Complete pressing time of the oxygen candles is equal to the sum of the holding time and pressing time, as shown in equation (4):

$$t_c = t_p + t_h \qquad (4)$$

where t_c is complete pressing time of oxygen candle, t_p is pressing time, and t_h is holding time.

Oxygen concentration can be determined using CD7, and the growth rate of oxygen concentration per second can be calculated by equation (5):

$$V_{X1\text{-}X2} = (C_{X2} - C_{X1}) / C_{X1} \qquad (5)$$

where $V_{X1\text{-}X2}$ is the growth rate of oxygen concentration from time X1 to time X2 (X2 - X1 = 1), and C_{X1} and C_{X2} are the oxygen concentrations at time X1, and X2, respectively.

In this study, U is defined as the stability coefficient of oxygen production (SCOP), and is expressed in equation (6):

$$U = \frac{2}{\pi}\arctan\frac{1}{|\,V_{X1-X2}-V_{X2-X3}\,|} \qquad (6)$$

A larger value of U indicates more stable performance of oxygen production. The value of U lies between 0 and 1.

3. RESULTS AND DISCUSSION

3.1 Dry pressing and wet pressing experiments

In the dry pressing and wet pressing experiments, pressure-application speed was 0.1 kN/s, the target pressure was 35 kN, holding time was 5 min, and the OCBs were dried at 60°C for 12 h. As shown in Figure 2, after drying, a number of irregular cracks appeared in the surface of wet-pressed OCBs with slight expansions in upper and lower bottom parts. The color of the wet-pressed OCBs was darker than that of dry-pressed OCBs. During wet pressing, surface temperature of the OCBs was increased, and trace amounts of chlorine was detected. Through compression strength experiments, the density and ultimate compressive strength (UCS) of W1 and W2 were found to be significantly higher than that of D1, as shown in Table 2. In addition, although the density was similar between W1 with W2, the UCS of W2 was higher than that of W1.

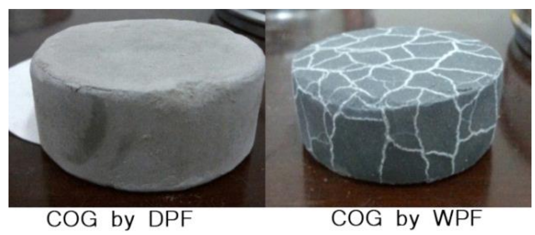


Figure 2: Dry-pressed and wet-pressed OCBs.

Table 2: Parameters of dry-pressed and wet-pressed OCBs.

Number	Pressure /kN	Pressurized speed /kN/s	Quantity /g	Highly /mm	Density × 10^{-3} /g·mm^{-3}	MCS /kN
D1	35	0.1	144.06	28	1.821	2.87
W1	35	0.1	145.04	22	2.333	24.81
W2	35	0.1	139.34	21	2.348	32.46

Note: D1 is the dry-pressed OCB; W1 is the OCB with 5 ml of added water; W2 is the OCB with 10 ml of added water.

The UCS of the wet-pressed OCBs was higher than that of dry–pressed OCBs. However, as constant heat is required for drying, the wet pressing process requires a longer time. In addition, the distribution of internal pores are heterogeneous after drying since it is difficult to mix the powder and water homogeneously, leading to differences in the amount of water inside the OCB. Homogeneous pores were produced after the water evaporated during the drying process. In addition, the powder appeared to react with water because the surface temperature of the aqueous powder was clearly increased and trace amounts of chlorine were detected. This is not only harmful but also reduces the effective oxygen occlusion amount and the performance of OCBs. Consequently, dry pressing is considered to have a better forming effect.

3.2 *Pressure-application speed and holding time experiments*

As shown in Table 3, the density of OCBs, pressed at different pressure-application speeds for a constant complete pressing time, showed a slight increasing trend with increases in pressure-application speed. The pressure-application speed had little effect on the density of the oxygen candle. As shown in Figure 3, the UCS of the three OCBs were 3.83 kN, 5.48 kN, 5.83 kN, and there was a positive correlation between the UCS of OCBs, pressure-application speed, and holding time.

Table 3: Parameters of OCBs in the pressure-application speed and holding time experiments.

Number	Pressurized speed/ KN/S	Pressure time /min	Holding time /min	Quality /g	Highly /mm	Density/$\times 10^{-3}$ $g \cdot mm^{-3}$
1	0.1	5.8	4.2	144.50	29.0	1.762
2	0.3	1.9	8.1	144.65	29.0	1.764
3	0.5	1.1	8.9	144.71	28.9	1.771

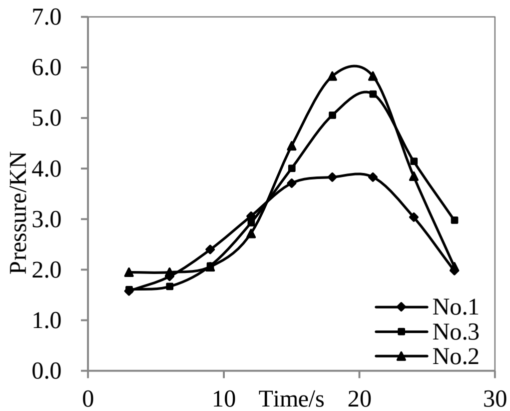


Figure 1: Experimental curves of compressive strength.

The results of the combustion experiment are shown in Figure 4. Oxygen concentration began to rise from 2 s and reached the highest at 4 s, and then it remained fluctuating at around 31%. Note that the main reaction occurred between 2–4 s. The growth rate of oxygen concentration was calculated, and the results are shown in Figure 5. According to the results, U1 = 0.966, U2 = 0.724, and U3 = 0.770.

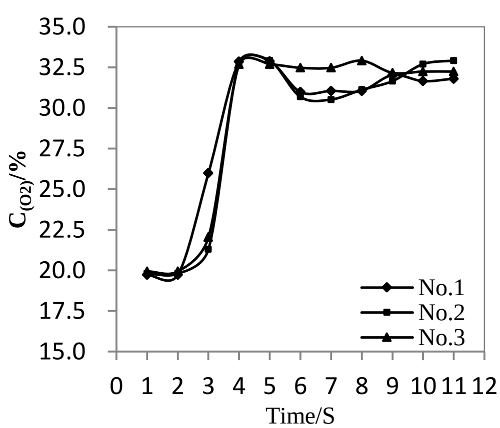


Figure 2: Oxygen concentration curve in a sealed chamber.

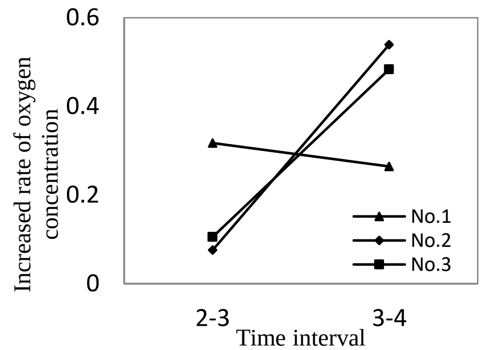


Figure 5: Velocity diagram of oxygen concentrations in a sealed chamber.

After the reaction was complete and the OCBs cooled to room temperature, the asbestos cloth wrapped around the OCBs were removed, and the OCBs were cut along the diameter, as shown in Figure 6. According to the observation of the sections, OCB No. 1 was completely burnt, and homogeneous holes were distributed across the burnt area in the shape of an upright cone. The burnt area of OCB No. 2 was small and distributed with heterogeneous holes. The combustion area of No. 3 was even smaller than that of No. 2 and was concentrated around the center of upper part; heterogeneous holes were also observed. In addition, caking was also observed in the right part of OCB No. 3.

During the entire process, not only did the shape of powder particles change, but the volume and density also changed constantly (Yu et al., 2000). In pressing processes, friction exists between the mold wall and powder particles, as well as between individual powder particles. The friction between powder particles increased when the pressure-application speed was increased, and consequently, powder flow ability was decreased. This resulted in increased pressure loss and heterogeneous distribution of density in the OCBs. This is the main reason for the larger and heterogeneous holes that occurred in the combustion area of OCBs No. 2 and No. 3 and also for the caking in the unburned area in OCB No. 3. In addition,

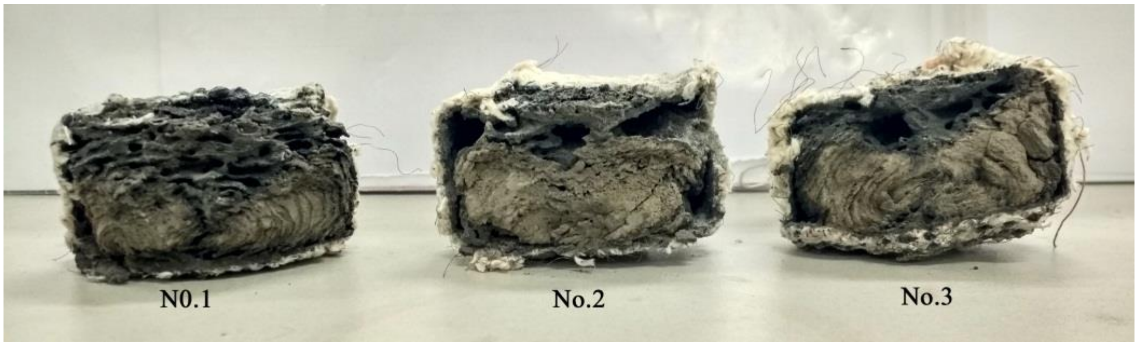


Figure 6: Photographs of OCBs after combustion

although a long holding time can enhance the physical properties of the OCBs, the air between powder particles is reduced, and the average distance between powder particles is decreased. As such, the effective area of combustion decreases, weakening the heat absorption capacity of unburned areas. The decomposition reaction of sodium chlorate powder stops without adequate heat, resulting in the large unburned areas in OCBs No. 2 and No. 3. According to the value of SCOP and observation of combustion residue of OCBs, the collating sequence of combustion stability is No. 1 > No. 2 > No. 3.

From the experiments, the following conclusion can be drawn: in dry pressing, the physical properties of oxygen candles can be improved by increasing the pressure-application speed and holding time. However, the rate of oxygen production is inconsistent, Furthermore, long holding times will increase the density of oxygen candles, reduce heat transfer capability, and eventually lead to a stop in the middle of the reaction.

4. CONCLUSIONS

Through dry pressing and wet pressing experiments, this study revealed that the dry pressing of oxygen candles has a better forming effect. The UCS and SCOP of the OCBs, pressed at pressure-application speeds of 0.1 kN/s, 0.3 kN/s, and 0.5 kN/s, were 3.83 kN, 5.48 kN, and 5.83 kN and $U_1 = 0.966$, $U_2 = 0.724$, and $U_3 = 0.770$, respectively. From the observation of combustion residue, it can be concluded that increases in the pressure-application speed, within an appropriate pressure range, decreases the stability of oxygen production.

Further research will focus on optimizing the pressing process of oxygen candles through quantitative studies on target pressure, pressure-application speed, and holding time.

5. ACKNOWLEDGEMENT

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6. NOMENCLATURE

OCB	oxygen candle block
UCS	ultimate compressive strengths
SCOP	stability coefficients of oxygen production
OSC	oxygen storage capacity
OSP	oxygen supply performance
t_c	complete pressing time
t_p	pressing time
t_h	holding time
V_{X1-X2}	growth rate of oxygen concentration from time X1 to time X2 (X2 - X1 = 1)
C_X	oxygen concentrations at time X

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