Research Article

The Oxygen Generation Performance of Hollow-Structured Oxygen Candle for Refuge Space

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To improve oxygen generation performance, we dissected and analyzed the incompletely reacted oxygen candles and thus proposed the concept of the hollow-structured oxygen candle. We calculated the surface area ratio and designed the mold for hollow-structured oxygen candles at a radius of 0, 5, 9, 12, 15.5, and 20 mm. The structural stability of the oxygen candles was tested by the loading experiment. The oxygen generation rate (OGR) and other properties were explored by combustion experiments. The composition of the oxygen candles and the residual solids after combustion were observed with scanning electron microscope (SEM). The results show that, with the increase of the hollow-structure radius (r), the stability of the hollow-structured oxygen candles gradually weakens, and the oxygen candles cannot be made when r is 20 mm. The hollow structure has a positive influence on the combustion of the oxygen candles, and the extent of combustion reaction can increase to 98.3% at the most, a 28.9% increase compared with that of solid-structured oxygen candles. The analysis of the morphology of cobalt(II,III) oxide and the macroscopic and microscopic morphology of the oxygen candles before and after combustion validates the rationality of the concept of the hollow-structured oxygen candle. The best performance of the oxygen candles is obtained when $r = 9$ mm, where the highest extent of combustion reaction is 98.3%. The total oxygen generated is 48.75 L, and the average rate is 9.2 L/min. The test of the oxygen candles with $r = 9$ mm after combustion by the X-ray diffractometer (XRD) shows that these oxygen candles fully reacted. This study provides a basis for optimizing the performance of oxygen candles and for the development of the oxygen supply system fitted with the oxygen candle in spaceflight, submarine, and underground space.

1. Introduction

Refuge space system can provide the people in distress with a living space, [1–3] which can isolate toxic and harmful gases and supply oxygen, water, food, and other necessary substances. It is installed with the oxygen supply system, carbon dioxide, carbon monoxide, climate control system, etc. [4]. The oxygen supply system is especially more important in the refuge space system [5, 6]. The common oxygen supply methods used in the refuge space include the forced air system, compressed oxygen cylinder, and chemical oxygen supply [7]. Among them, the chemical oxygen supply commonly includes water-electrolyzed, potassium hyperoxide, peroxide, and oxygen candle. Similar to the liquid oxygen density, the oxygen candle is known for

lightweight, safety, high oxygen storage capacity, and a long shelf life and thus has been extensively used in the internal life support systems of protective engineering, submarine, civilian aircraft, and fire-fighting [8–11]. Moreover, its oxygen generation performance is not influenced by surrounding temperature and humidity. Hence, it has become the common source of chemical oxygen supply.

Scholars worldwide mainly focused on the oxygen candle formula, catalyst, fuel, burning fluctuation, size of the composition, start-up mode, and molding process which influence the performance of oxygen candles [12–16]. Our team has contributed to the performance of oxygen supply in refuge space for years [17–20]. We concentrated on the oxygen candle formula, catalyst, molding process, and start-up mode by thermogravimetric analysis, orthogonal

experiment, and other laboratory experiments. These findings could provide scientific basis for continuous optimization of the oxygen candle. However, few studies focused on the oxygen generation performance of the oxygen candle with different structures. In this paper, according to the dissection and analysis of the incompletely reacted oxygen candle, we put forward the concept of the hollow-structured oxygen candle. Furthermore, we perform laboratory studies to explore the oxygen generation performance of the hollow-structured oxygen candle, investigate the micromorphology of the oxygen candles before and after combustion using SEM, test the chemical composition of solid combustion products by XRD, and describe their influence on the combustion of the oxygen candles. This study can benefit the development of the oxygen candle system in spaceflight, submarine, and underground space.

2. Hollow-Structured Oxygen Candle

2.1. Design of the Hollow-Structured Oxygen Candle. The principle of oxygen generation of the oxygen candle is as follows: the oxygen generation reagent, sodium chlorate, decomposes to generate oxygen at high temperature produced by the combustion of metal fuel Mn powder. Two chemical equations are mainly involved:

The previous experiments showed that some oxygen candles made even by the optimal formula [19] cannot be completely combusted, as shown in Figure 1. The simple dissection of these oxygen candles reveals that the top and the periphery are fully combusted, and the unreacted parts are distributed inside the oxygen candles, near the oxygen candle axis. So, the closer the parts to the surface, the fuller they are combusted. The reason is that the substances accumulated in the external reaction hinder the heat transfer to the inside.

Actually, in the reaction, Mn powder is used as a fuel that maintains burning. Therefore, the complete reaction of the oxygen candles mainly depends on whether the Mn powder can fully combust. The combustion of Mn powder is a gas-solid two-phase reaction. In this case, when combustion occurs, the combustion products are continuously generated from the combustion zone, while the fuel enters the combustion zone. Otherwise, the combustion will not sustain. In this process, the departure of the products and the entry of the fuel involve the transfer of materials (gas, heat, etc.) [21, 22]. So, the increase of channels for the material transfer allows complete reaction of Mn powder in the oxygen candle. In addition, the gas-solid two-phase combustion reaction in combustion and heat-transmission science discovered that the specific surface of solid particles, which influences the rate and extent of the reaction, provides a contact surface and reaction area for combustion reaction.

Subsequently, we propose the concept of the hollow-structured oxygen candle [10]. The hollow structure adds the

FIGURE 1: Incompletely reacted oxygen candles.

transmission channels of gas and heat, allowing the heat to diffuse as evenly as possible to the unreacted part of the oxygen candle and to preheat the parts to be combusted in no time. Besides, the hollow structure can increase the surface area of the oxygen candle and provide the necessary contact surface and effective reaction area in the combustion reaction. The schematic diagram is shown in Figure 2.

2.2. Determination of the Hollow Structure Size. According to the results of previous studies and the existing pressing process of the oxygen candle [17, 19], we determine that the mass of OCB is 200 g; correspondingly, the height of the pressed OCB is about 40 mm. Under the same stress, loading rate, and holding time, the density of the solid-structured OCB ρ_1 is equal to that of the hollow-structured OCB ρ_2 . We control the mass M of the two OCBs to be the same and the maximum radius R of the two OCBs to be the same, at a constant of 30 mm. Thus, the volume of the solid-structured OCBs (V_1) and the hollow-structured OCBs (V_2) is the same. We perform the following calculation based on this premise, and then the surface area ratio of the hollow-structured OCB to the solid-structured OCB is ΔP :

$$\Delta P = \frac{S_2}{S_1} = \frac{2\pi(R^2 - r^2) + 2\pi h_2(R + r)}{2\pi R^2 + 2\pi R h_1}, \quad (3)$$

where S_1 and S_2 are the surface area of the solid- and hollow-structured OCBs, respectively; r is the radius of the hollow structure; and h_1 and h_2 are the height of the solid- and hollow-structured OCBs. Then, we can know the relationship between ΔP and r , as shown in Figure 3, which shows that with the increase in r , the surface area ratio slowly increases before $r = 20$ mm and then sharply increases. When r is close to 30 mm, the surface area ratio tends to infinity. According to the actual situation of the experiment, we select the hollow-structured OCBs with the following property as research objects: the ΔP of which is equal to 1.0, 1.1, 1.2, 1.3, 1.5, and 2.0 and the theoretical values of r are equal to 0, 5, 9, 12, 15.5, and 20 mm, respectively.

3. Experiment

3.1. Manufacture and Compression Test of Hollow-Structured OCBs. We first weighed the reagent in proportion (Figures 4(a)–4(d)) according to the optimal formula of the oxygen candles and then mixed them using a KE-2L planetary ball mill (with a metal grinding jar and grinding

FIGURE 2: Schematic of heat transfer and oxygen release of solid-structured oxygen candles (a) and hollow-structured oxygen candles (b).

FIGURE 3: Relationship between ΔP and r .

ball) and set the speed at 280 r/min and the time at 5 min. The well-mixed powder (Figure 4(e)) was placed in a vacuum-drying oven to be dried at 60°C for 12 hours, and then the dried powder was poured into the mold (Figure 4(f)) and placed on a press to manufacture the OCBs (Figure 4(g)). In the experiment, the YES-300 press and a self-designed mold were employed. The fast loading rate was 0.5 kN/s, and holding time was 5 minutes. We put the OCBs on the press to test their axial compressive strength and radial compressive strength (Figures 4(h) and 4(i)) and then judged their structural stability.

3.2. Oxygen Generation Experiment of Hollow-Structured Oxygen Candles. The periphery of OCBs was wrapped in an insulating material (ceramic fiber) (Figure 4(j)). The small ignition head was fixed on the OCBs (Figure 4(k)), and 3 g ignition powder was used to cover the small ignition head and the upper surface of the OCBs. Then, the insulating material was used to seal the oxygen candle in order to prevent heat dissipation, maintain the reaction, and ensure that the small ignition head and ignition

powder are in close contact when the small ignition head is ignited (Figure 4(k)). Next, we placed the oxygen candle in the reaction vessel and put them together into a 425 L sealed box. We connected the wire of the small ignition head with the igniter and opened the gas detector and temperature logging device. In the end, we closed the door of the sealed box and started the igniter by a remote control.

In this experiment, the oxygen concentration was recorded using the CD-KJ70N monitoring system, which is designed on the basis of the “Coal Mine Safety Regulations” [23] and the actual conditions of underground mines and can collect the environmental parameters such as oxygen concentration, carbon dioxide concentration, carbon monoxide concentration, gas concentration, wind speed, and air pressure. The monitoring system has obtained the coal safety mark and explosion-proof qualification certificate. A wireless, remotely controlled igniter (A01; Liuyang Fireworks Development Co., Ltd., China), composed of a receiver box and remote-control unit, was used to start the ignition. The temperature of the reaction vessel was detected in real time using a temperature-monitoring instrument (Beijing Sailing Technology Co., Ltd., China), which consisted of an STI-AS temperature-inspecting instrument (measurement accuracy: 0.2% FS) and a PC1625 platinum resistance temperature detector (PRTD) (operating temperature: -200 to 600°C; error: $\pm 0.06\%$). The PRTD was fixed outside the reaction vessel. The experimental data were collected using the computer software and recorded in real time.

3.3. SEM Experiments. We collected the reagent that made up the oxygen candle, some well-mixed oxygen candle powders, and the small particles with fresh section of the OCBs after combustion for the SEM experiment. ZEISS Sigma SEM was employed to research the micromorphology of oxygen candles before and after combustion. Its secondary electron image resolution can reach 1.3 nm (20 kV), the acceleration voltage of the electron source can be adjusted from 100 V to 30 kV, and the magnification range is 12x to 1000,000x.

FIGURE 4: The process of experiments: (a) sodium chlorate; (b) Mn powder; (c) cobalt(II,III) oxide; (d) kaolin; (e) well-mixed powder of the oxygen candles; (f) mold manufactured; (g) oxygen candles at different r ; (h) axial compressive strength test; (i) radial compressive strength test; (j, k) wrapping of the OCBs; (m, n) the oxygen candles after combustion.

Figure 4 illustrates the flow diagram of the process of the experiments.

4. Analysis of the Results

4.1. The Maximum Load for Ejection. In the manufacture process, we found that the OCBs at $r = 0, 5, 9, 12$, and 15.5 mm can be made and the OCBs at $r = 20\text{ mm}$ failed to be made. After five tries, all OCBs at $r = 20\text{ mm}$ cracked, as shown in Figure 5. The reason attributes to the following: (1) at $r = 30\text{ mm}$ and $r = 20\text{ mm}$, the effective thickness of the OCBs is only 10 mm . Thus, the structure is unstable. (2) The ejection process is not continuous, which is prone to create stress concentration and physical deformation due to instantaneous release, as shown in the dotted line frame in Figure 5(b). So, we did not study the OCBs at $r = 20\text{ mm}$.

Figure 6 indicates that when $r \leq 9\text{ mm}$, the maximum load for ejection slowly increases with the increase of r . When $r \geq 9\text{ mm}$, the load for ejection sharply increases as r increases. By comparative analysis, we can find that the larger load ejection required causes the greater damage and difficulties in manufacturing the OCBs. The larger the load for ejection, the bigger the stress concentration and

FIGURE 5: Oxygen candle at $r = 20\text{ mm}$.

physical deformation due to the instantaneous release. Thus, the large load for ejection is not conducive to the manufacture of OCBs.

4.2. The Maximum Compressive Strength. Figure 7 shows that the maximum axial compressive strength of each OCB is greater than the maximum radial compressive strength, and

FIGURE 6: Maximum load for ejection on manufacturing hollow-structured OCBs.

FIGURE 7: Maximum axial compressive strength and maximum radial compressive strength of hollow-structured oxygen candles.

the maximum axial compressive strength and maximum radial compressive strength decrease with the increase in r . At $r = 0, 5$, and 9 mm, the reduction of the maximum axial compressive strength is not obvious. At $r = 12$ and 15.5 mm, the maximum axial compressive strength sharply decreases, and the maximum axial compressive strength of the hollow-structured OCBs at $r = 15.5$ mm is only 31.8% of that at $r = 0$ mm. At $r = 0$ and 5 mm, the reduction of the maximum radial compressive strength is not obvious. At $r = 9, 12$, and 15.5 mm, the reduction of the maximum radial compressive strength is significant, and the maximum radial compressive strength of the hollow-structured OCBs at $r = 15.5$ mm is only 8.2% of that at $r = 0$ mm.

The result of this experiment can be summarized as follows: with the increase in r , the stability of the OCB structure decreases.

4.3. Variation of Oxygen Concentration with Time. Figure 8 illustrates the change of oxygen concentration of each oxygen candle. In the first minute, the oxygen concentration fluctuates slightly, and then it starts to increase dramatically. After a slight decrease, there is an approximate line increase over time, and finally, it reaches a maximum value.

The data in Figure 8 expound that the final concentration of the oxygen candles at $r = 0, 5, 9, 12$, and 15.5 mm is 28.8%, 31.43%, 32.05%, 32.1%, and 32%, respectively, indicating that at $r = 0$ and 5 mm, the reaction extent of the oxygen candles is lower, and at $r = 9, 12$ and 15.5 mm, the extent is higher. In this figure, the time for each curve to reach the maximum value is different, which is 400, 400, 360, 340, and 300 s at $r = 0, 5, 9, 12$, and 15.5 mm, respectively. We can conclude that, with the increase in r , the reaction extent of the oxygen candles increases and the reaction rate accelerates.

The above results show that the hollow structure has a positive influence on the oxygen generation performance of the oxygen candles.

4.4. The Total Oxygen Generation. Table 1 indicates that, with the increase in r , the oxygen generation displays an increasing trend. The difference in the total volume of oxygen generation of the oxygen candles at $r = 9, 12$, and 15.5 mm is very small. The ratio of actual volume to theoretical volume at $r = 9, 12$, and 15.5 mm is close to 100%, and the difference does not exceed 3%, demonstrating that the reaction of the oxygen candles at $r = 9, 12$, and 15.5 mm is more complete, and the highest total oxygen generation is 48.75 L at $r = 9$ mm.

4.5. The Average Rate of Oxygen Generation. The average OGR of the oxygen candles at different r is calculated, as listed in Table 1, which indicates that with the increase in r , the average OGR accordingly increases. So, the hollow structure in the oxygen candles has a positive influence on the oxygen generation; namely, within a certain range, the larger the r , the less the unreacted part of the oxygen candles.

4.6. The Temperature of the Reactor Vessel. Figure 9 exhibits that the temperature of the reactor vessel for each oxygen candle displays a sharp increase at the beginning and then slowly rises. When it reaches the highest value, it begins to slowly decline. In addition, the rate of temperature rise increases as r increases, and the maximum temperature increases accordingly. At $r = 0$ mm, the oxygen candle generates a little heat, leading to a small value of temperature; correspondingly, the maximum temperature is the lowest among the 5 hollow-structured oxygen candles. This indicates that the reaction of this oxygen candle is the least. At $r = 15.5$ mm, the rate of temperature rise is the fastest, and the maximum temperature is the largest, which reveals that the reaction of this oxygen candle is more complete and the rate is more rapid. The temperature difference in the reactor vessel before and after the reaction correspondingly increases with the increase in r , which shows the consistent variation of temperature.

FIGURE 8: Relationship between oxygen concentration and reaction time.

TABLE 1: Oxygen concentration, oxygen generation, and the average OGR.

Radius of the hollow structure (mm)	The initial oxygen concentration (%)	The oxygen concentration at the end of reaction (%)	The reaction time (min)	The actual volume (L)	The theory volume (L)	The ratio of actual volume to theoretical volume (%)	The average OGR ($\text{L} \cdot \text{min}^{-1}$)
0	20.7	28.8	6	34.43	49.61	69.4	5.74
5	20.62	31.43	5.7	45.94	49.61	92.6	8.06
9	20.58	32.05	5.3	48.75	49.61	98.3	9.2
12	20.6	32.1	5	48.45	49.61	97.7	9.69
15.5	20.62	32	4.3	48.36	49.61	97.5	11.25

FIGURE 9: Relationship between the temperature and time.

4.7. *The Macroscopic and Microscopic Morphology of Oxygen Candles before and after Combustion.* Figure 10 shows the macroscopic and microscopic morphology of the oxygen candles before and after combustion. It can be observed from Figures 10(a) and 10(b) that most of the cobalt (II, III)

oxide exists in the spheres or ellipsoids at a radius of about 5–10 μm , whose morphology is like a flower cluster to be unfolded and has more pores. These pores increase the specific surface area of the cobalt (II, III) oxide and become a pathway for heat and oxygen transport, promoting the combustion reaction. As shown in Figures 10(c) and 10(d), the burned oxygen candles display a porous structure, where some pores penetrate the oxygen candles (red circle portion) and some ones are interconnected (yellow circle portion). Figure 10(e) displays that there are many small holes (red circle portion) with different sizes in the fresh section of the burned oxygen candles, which extend inward. There are also some large pits (yellow circle portion) and small holes in the pits as well. The presence of these small holes and large pits allows heat and oxygen to be transferred and exchanged during the reaction and facilitates the combustion reaction. The above research further validates the rationality of the concept of the hollow-structured oxygen candle proposed in this paper.

5. Determination of the Best Performance of Hollow-Structured Oxygen Candles and Verification by the XRD Experiment

5.1. *Determination of the Best Performance.* Because the oxygen candle at $r = 0$ mm has low extent of reaction, we will not further discuss it. We focus our discussion on oxygen

FIGURE 10: The macroscopic and microscopic morphology of the oxygen candles before and after combustion. Microscopic morphology of cobalt(II,III) oxide (a) and its local enlargement (b) by SEM; macroscopic morphology of the oxygen candles after combustion (c, d); microscopic morphology of fresh section of the oxygen candles after combustion (e) by SEM.

candles at $r = 5, 9, 12$, and 15.5 mm, which have high extent of reaction. To determine the best performance of hollow-structured oxygen candles, we analyze the results of the above experiments synthetically. On the basis of four indicators, compressive strength, oxygen generation, average OGR, and the temperature of the reactor vessel, we assign a number to the oxygen candles at different r and identify the weight of the four indicators according to their importance in engineering practices [17–19], as listed in Table 2. The comprehensive score of the oxygen generation performance = $\sum \text{score of index} \times \text{weight}$. Then, we obtain the comprehensive score of each oxygen candle at different r , as listed in Table 2. So, we determine that the oxygen candle at $r = 9$ mm has the best performance in terms of oxygen generation, and the axial compressive capacity and radial compressive capacity are 2.89 kN and 1.22 kN, respectively. Its oxygen generation is 48.75 L, the average OGR is 9.2 L/min, and the temperature of the reaction vessel is 79.4°C .

5.2. Analysis of the XRD Experiment. We collected the residual solids of the oxygen candles with $r = 9$ mm after combustion and grinded them into powder for XRD analysis. X-ray diffraction analysis was carried out on a Bruker D8 Focus diffractometer, whose light source is radiation from a copper target of an X-ray tube. The tube voltage is 40 kV, the tube electricity is 30 mA, the scanning angle is from 20 to 120° , and the step width (relative to 2θ) is 0.02° , used to test the chemical composition of the oxygen candles after combustion.

It can be seen from Figure 11 that the main compositions of the hollow-structured oxygen candle with $r = 9$ mm after combustion are halite (sodium chloride), hausmannite (manganese tetroxide), manganosite (manganese oxide), and cobalt (II, III) oxide. The results show that all the sodium ions in the combustion products exist in the form of sodium chloride. There are two kinds of combustion products of Mn powder, namely, manganese oxide and manganese tetroxide. The existence of manganese oxide indicates that some Mn powder does not react completely to form a stable compound manganese dioxide. The existence of manganese tetroxide indicates that oxygen produced a violent oxidation reaction of some manganese powder. Because the kaolin is mainly composed of aluminum oxides, silicon oxides, and crystalline water, the relative molecular mass is large, resulting in less aluminum and silicon content, so we did not detect the existence of aluminum oxides and silicon oxides. The above test and analysis results show that the hollow-structured oxygen candle with $r = 9$ mm is completely reacted.

6. Conclusions

We have dissected and analyzed the solid-structured oxygen candles after combustion and proposed the concept of the hollow-structured oxygen candle. According to the laboratory experiment results, we drew the following conclusions:

TABLE 2: The number, the weight, and the comprehensive score of each indicator.

Index	$r = 5 \text{ mm}$	$r = 9 \text{ mm}$	$r = 12 \text{ mm}$	$r = 15.5 \text{ mm}$	Weight
The compressive strength	4	3	2	1	0.2
The oxygen generation	1	4	3	2	0.5
The average OGR	4	3	2	1	0.2
The temperature of the reactor vessel	4	3	2	1	0.1
The comprehensive score	2.5	3.5	2.5	1.5	—

FIGURE 11: The results of the XRD experiment.

- (1) With the increase in r , the compressive strength of the hollow-structured oxygen candle decreases. The lower the compressive strength, the poorer the structural stability, which is adverse for manufacturing oxygen candles. When $r = 15.5 \text{ mm}$, the axial compressive capacity and radial compressive capacity of the hollow-structured oxygen candle account for 31.8% and 8.2% of those of the solid-structured oxygen candle, respectively. And when $r = 20 \text{ mm}$, the oxygen candle cannot be formed.
- (2) The hollow structure is beneficial in improving the performance of the oxygen candle. In our experiments, the hollow-structured oxygen candle can increase the oxygen generation ratio to 98.3%, an increase of 28.9% compared with that of the solid-structured one.
- (3) A comprehensive analysis of the four indicators shows that the oxygen candle at $r = 9 \text{ mm}$ has the best performance of oxygen generation among the six sizes of hollow-structured oxygen candles. Its axial compressive strength and radial compressive strength are 2.89 kN and 1.22 kN, respectively, and the total oxygen generated is 48.75 L. The average OGR is 9.2 L/min, and the highest temperature of its reactor vessel is 79.4°C.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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