Coupling Analysis of Chemiluminescence and Pressure Changes in CH₄/Air Explosion Suppressed by NaHCO₃ Powder

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ABSTRACT: This paper presents the coupling relationship between the flame emission spectrum and explosion characteristics of CH₄/air mixtures with NaHCO₃ powder added. Due to the test of different concentrations of suppression powder in 20 L spherical-explosion-test devices, the flame emission spectrum and explosion pressure data were collected. In this experiment, four kinds of excited radicals, including CN*, HCHO*, CO*, and OH*, have a higher probability of being detected, and the changes of their existence duration and spectral intensity show strong regularity. Results reveal that for suppressant concentration in the range of 0−50 mg/L, together with the improvement of a suppression effect, the existence duration and spectral intensity of CN*, CO*, and OH* decrease, which is opposite to that of HCHO*. Besides, the spectral intensity of OH* and CO* shows a good linear relationship with the change of the maximum explosion pressure. Controlling the content of CN*, CO*, OH*, and HCHO* is of great significance in suppressing the explosion.

1. INTRODUCTION

A gas explosion is one of the most serious accidents in coal mines. CH₄ is the major ingredient of mine gas. The mitigation of gas explosion hazards is an important topic in the process safety industry.

Among the current major explosion suppressants, powder explosion suppressants have gradually become the preferred choice for suppressing methane explosion due to their advantages of high efficiency, low cost, easy preservation, and safe transportation.¹ Some powder fire extinguishing agents that have shown good fire extinguishing efficiency have been widely studied due to their inhibition effect on methane explosion, such as various dry chemicals, SiO₂ and its homogeneous substances, and urea. NaHCO₃ is the main component of the BC dry powder extinguishing agent, which has become one of the main research objects due to its good effect in extinguishing combustible liquid and gas fires. At the same time, the development of a flame emission spectroscopy study makes flame chemiluminescence an important means of studying combustion and explosion.

Earlier research on NaHCO₃ powder mainly focused on its suppression effect on combustion or explosion. The flame suppression effects of NaHCO₃ are produced by both physical and chemical processes.²−⁴ The chemical process means that the powder directly participates in after gasification or produces new substances after pyrolysis to participate in the methane oxidation reaction, which interferes with the chain reaction.⁴−¹¹ At the beginning, researchers compared the flame suppression effect of NaHCO₃ and other various sodium salts⁵ and believed that the flame suppression mechanism of sodium salt was consistent and Na could inhibit the production of OH*. With further studies, the gaseous NaOH produced after the pyrolysis of NaHCO₃ is considered to be the main...
NaOH inhibits the chain transfer and chain branching process by consuming a large amount of OH radicals. In comparison, the chemical process plays a more important role in suppressing combustion. At least 50% of the fire extinguishing efficiency depends on chemical processes, which is why reducing the powder particle size can improve the efficiency of fire extinguishing. At the same mass concentration, the reduction in particle size will increase the specific surface area of the particles, which shorten the gasification time of the powder and increase the concentration of the gas-phase inhibitor in the combustible gas mixture per unit time. Thereby, the chemical suppression is enhanced. For micron-level NaHCO₃ powder, when the particle size of the powder is reduced to 20 μm, the fire suppression and explosion suppression performance can be improved by about 8−10 times. The experimental and simulation studies of Fan et al. showed that when the particle size of the powder is large, the physical endothermic effect plays a major role in inhibition. On the contrary, the chemical process plays a leading role. When the volume fraction of NaOH in the flame reaches about 0.003, the mass fraction of H and OH radicals in the flame drops to a stable value, and the laminar combustion rate will be significantly reduced.

At first, flame chemiluminescence was mainly used for a combustion diagnosis. The chemiluminescence of alkane−air flame mainly comes from OH* (280−310 nm), CH* (430 and 390 nm), C₂* Swan bands (mostly between 470 and 550 nm), CO₂* continuous bands, and HCO* (300−500 nm). It was found that partially excited particle chemiluminescence changes have a strong relationship with the equivalence ratio, flame temperature, heat release rate, and other combustion state parameters. Skaggs studied the change of OH* chemiluminescence under Fe(CO)₅, CF₃Br, and N₂ inhibition conditions first. He pointed out that the production of OH* will decrease with the increase of inhibitor addition, which proves the feasibility of studying combustion inhibition by flame chemiluminescence. Then, Liu studied the luminence characteristics of OH* and CH* in the methane flame under the action of water mist and found that the emission spectrum intensity decreased with the increase of the water mist load ratio of the flame front. Yang first observed and measured the characteristic spectrum of the methane flame under the action of powder inhibitor through a camera device and a spectrum testing instrument. With the addition of four kinds of suppressants including fumed silica, ultrafine ABC dry powder, sodium bicarbonate, and potassium bicarbonate powders, he observed and analyzed the change of the free radical characteristic spectrum in the flame. It was found that all four inhibitors can suppress the production of OH* and CH*. However, there is a certain difference between the study of explosive flame luminescence and stable flame luminescence. Some elementary reaction rates will change due to the high temperature and high pressure during the explosion process, resulting in changes in the signal of the radiation characteristic curve. Li et al. used a spectrometer imaging system and high-speed camera technology to carry out a large number of experiments on pure methane explosion and explosion with different powder explosion inhibitors and found that in addition to CH* and OH*, CN*, HCO*, other intermediate particles have a high possibility of occurrence. Moreover, the spectrum intensity changes of radicals above are believed to be associated with the macroscopic explosion.

![Figure 1. Typical spectrum curve of explosion flame suppressed by NaHCO₃: (a) 180−420 nm, (b) 550−700 nm, and (c) 700−900 nm.](image-url)
suppression effects, such as maximum pressure and maximum pressure rise rate. They found that the process of pressure rising and changes of spectral intensity were synchronized.\textsuperscript{31} Luo and her team have studied the deflagration, pressure, and flame characteristics of methane or mixed gas in a closed vessel\textsuperscript{32−34} and the coupling relationship between the explosion pressure and explosive intermediate.\textsuperscript{35} She used the time difference between the peak explosion pressure and the peak CH\textsubscript{2}O concentration to measure the strength of their relationship. The coupling relationship has also been confirmed in the experimental study by Su.\textsuperscript{36} However, it has not been quantitatively analyzed, and the strength of the correlation between different particles and macroparameters has not been determined.

In summary, the NaHCO\textsubscript{3} powder has a good suppression effect on methane combustion, and the chemiluminescence of certain excited radicals in the flame is significantly related to the combustion states of the flame. However, at present, chemiluminescence is mostly used in stable flame studies and rarely applied to explosion suppression studies. Moreover, the relationship between the macroscopic explosion suppression effect and the excited radicals with high occurrence frequency except CH* and OH* is not well understood.

In this paper, the experimental method was used to study how the spectrum intensities of excited radicals including CN*, OH*, CH\textsubscript{2}O*, and CO* change with the concentration of NaHCO\textsubscript{3}. At the same time, the coupling relationship between explosion pressure and spectrum intensity was analyzed. This research can lay a foundation for further research on the relationship between the macroscopic explosion suppression effect and the microscopic explosion suppression mechanism and can be used as a reference for targeted performance improvement of explosion suppressant.

2. RESULTS AND DISCUSSION

2.1. Duration of Excited Radicals. Since the effect of suppressant is the strongest at the initial stage of explosion, only the spectral signal from the beginning of the explosion to the time when explosion pressure reaches the maximum is analyzed. As shown in Figure 1, through the analysis of the spectral peaks, nine kinds of excited radicals tend to be detected, including C\textsubscript{2}*\textsuperscript{37}, CH*, CN*, HCO*, CO*, OH*, HCHO*, H\textsubscript{2}O*, and CO\textsubscript{2}*. Figure 2 classifies the nine particles according to their existence duration changing. With the increase of the concentration of the explosion inhibitor, curves of OH* and CO* have a significant downward trend at the initial stage of explosion while that of HCHO* has an apparent extension trend. The remaining five kinds of particles do not have an obvious regularity.

When the concentration of NaHCO\textsubscript{3} increased from 0 to 12.5 mg/L, the existence duration of OH* and CO* decreased greatly for about 50% of the overall decrease, which means that they are sensitive to the suppressor. However, the existence duration of CN* and HCHO* was relatively stable. When the concentration of inhibitor further increased, the duration of OH* and CO* continued to decrease but they tended to become stable, while the duration of CN* and HCHO* began to change at a relatively steady rate. So, NaHCO\textsubscript{3} may play a role in inhibiting the generation of OH*, CN*, and CO* particles or accelerating their consumption. However, it has an opposite effect on

![Figure 2](http://pubs.acs.org/journal/acsodf/article/ACS_Omega_2020, 5, 27133−27141)

Figure 2. Variation in the duration of various particles under different NaHCO\textsubscript{3} concentrations. (a) Duration of OH*, CN*, and CO*. (b) Duration of HCHO*. (c) Duration of C\textsubscript{2}*\textsuperscript{37}, CH*, H\textsubscript{2}O*, CO\textsubscript{2}*, and HCO*.

![Figure 3](http://pubs.acs.org/journal/acsodf/article/ACS_Omega_2020, 5, 27133−27141)

Figure 3. Spectral intensity of CN* changes with different concentrations of NaHCO\textsubscript{3}. (a) Spectral intensity at 388.34 nm. (b) Spectral intensity at 415.81 nm. (c) Spectral intensity at 585.82 nm.)
HCHO*, OH* and CO* are more sensitive to NaHCO₃. NaCHO₃ may participate preferentially in elementary reactions involving OH* and CO* under low concentration conditions.

2.2. Relative Content Change of Particles. In fact, the spectral intensity signal cannot be detected at every characteristic wavelength because of the instability of explosion flame, measuring errors, and other reasons. So, we had to choose the relatively stable signal to analyze.

Figure 3 shows how the spectral intensity of CN* changes with the concentration of NaHCO₃ at three characteristic wavelengths: 388.34, 415.81, and 585.82 nm. When the concentration of NaHCO₃ increases from 0 to 60 mg/L, the overall trends of the spectral intensity are basically the same at three characteristic wavelengths. When no inhibitor is added, the spectral intensity is the largest and then it gradually decreases with increasing the concentration of inhibitor. The inflection point appears when the concentration of inhibitor reaches 50 mg/L, and the spectral intensity begins to rise. It can be considered that the content of CN* decreases with increasing the concentration of inhibitor. When the concentration of inhibitor reaches 50 mg/L, the inhibitory effect on CN* is the strongest.

Figure 4 shows how the spectral intensity of HCHO* changes with the concentration of NaHCO₃ at three characteristic wavelengths: 354.4, 384.65, and 457.3 nm. Although there is a sharp increase of spectral intensity at 384.65 nm, exceeding the spectral intensity of 12.5 and 60 mg/L when no inhibitor is added, the spectral intensity at the three wavelengths shows an increasing trend. When the concentration of inhibitor reaches 50 mg/L, the spectral intensity begins to decrease. The content of HCHO* is the highest under the condition of 50 mg/L inhibitor concentration and is the lowest under the condition of 0 mg/L inhibitor concentration, while the content under the other three conditions is not much different. Therefore, the inhibitor...
may suppress some elementary reactions, which causes the accumulation of HCHO$^*$. Figure 5 shows how the spectral intensity of CO$^*$ changes with the concentration of NaHCO$_3$ at three characteristic wavelengths: 215.02, 223.83, and 233.8 nm. Each spectral intensity has the same trend. They decrease to their minimum and increase gradually. The inflection point occurs at 50 mg/L. The addition of NaHCO$_3$ inhibits the conversion from intermediate products to CO$^*$ and lowers its content.

Figure 6 shows how the spectral intensity of OH$^*$ changes with the concentration of NaHCO$_3$ at three characteristic wavelengths: 281.6, 296.24, and 318.47 nm. The spectral intensities at three wavelengths showed a decreasing trend within the concentration range of 0−50 mg/L and started to rise when the inhibitor concentration increases further. The addition of NaHCO$_3$ may directly react with OH$^*$ or catalyze the related elementary reactions, which accelerates the consumption of OH$^*$ or inhibits the generation of OH$^*$.

2.3. Coupling Analysis of the Flame Emission Spectra and Explosion Pressure. To explore the relationship between the changes of four excited radicals and the macroscopic explosion suppression effect, the existence duration and spectral intensity and maximum explosion pressure were compared and analyzed. As shown in Figure 7, when the concentration of the depressant reaches 50 mg/L, $P_{\text{max}}$ drops to the lowest point. It can be approximately considered that when the inhibitor concentration is 50 mg/L, the oxidation of methane is suppressed to the greatest extent. With the concentration of NaHCO$_3$ going up, the existence duration and spectral intensity of the four particles showed a good correlation with the change of the maximum pressure. Among them, the existence duration and spectral intensity of CN$^*$, CO$^*$, and OH$^*$ showed a decreasing trend with increasing $P_{\text{max}}$, while that of HCHO$^*$ showed an increasing trend. Moreover, the spectral intensity curve has an inflection point at a concentration of 50 mg/L, which is different from the existence duration curve. In comparison, the spectral intensity is better coupled to the explosion pressure. Figure 8 illustrates the linear fitting curves. The $R^2$ of CO$^*$ and OH$^*$ were all above 0.9, which indicates that there was a strong linear relationship.

2.4. Discussion. From the above results, it can be discovered that the changes of excited radicals and explosion pressure are synchronous apparently. Especially for spectral intensity and maximum pressure, the linear relationship is stronger. Based on the present suppression mechanism study, NaHCO$_3$ mainly reduces the content of OH$^*$ and O$^*$. Since OH$^*$ plays the most important role in the process of chain transfer and chain branching, many elementary reactions can be inhibited. That is the reason why some excited radicals’ content changes. For example, according to the study of the flame emission spectrum in a shock tube, OH$^*$ is mainly produced through the following reaction.

Figure 7. Coupling diagram of maximum explosion pressure, free radical duration, and spectral intensity in different concentrations of NaHCO$_3$: (a) CN$^*$, (b) HCHO$^*$, (c) CO$^*$, and (d) OH$^*$.
The production of CH₄ is closely associated with OH. The reduction of OH indirectly inhibits the production of OH*. Actually, the mechanism of explosion suppression is pretty complicated, so is the study of new suppressant. Now, it may be simplified using the linear relationship between spectral intensity and explosion characteristics, such as maximum pressure. The spectral intensity of OH* and CO* can be used to measure the antireexplosion effect, which can help make targeted improvement on a suppressant.

3. CONCLUSIONS

The existence duration and spectral intensity of the CH₄ explosion flame were analyzed with different concentrations of NaHCO₃ powder added. Results indicate that 50 mg/L is the best concentration to suppress methane explosion, and there is an obvious change rule and some excited radicals and explosion pressure changes were coupled. (1) CN*, HCHO*, CO*, and OH* are the key excited radicals in the methane explosion process. The existence duration of OH*, CN*, and CO* in the initial stage of the explosion has a clear decreasing trend and that of HCHO* indicates a significantly increasing trend. When the concentration of the inhibitor is low, the existence duration of OH* and CO* decreases rapidly, and as the concentration further increases, the decreasing rate slows down. The content of CN* and HCHO* does not change significantly at low inhibitor concentration but starts to change at a stable rate when the concentration rises. OH* and CO* are more sensitive to NaHCO₃ and more susceptible to interference. (2) With the enhancement of the explosion suppression effect, the relative contents of CN*, CO*, and OH* show a downward trend, while for HCHO*, the relative contents show an upward trend. When the explosion suppression effect begins to decline, the above particles will show the opposite trend. The spectral intensity of OH* and CO* has a strong linear relationship with the explosion suppression effect. They can be used to measure the effect of suppressants, and controlling their content is of great significance in explosion suppression.

4. EXPERIMENTAL AND ANALYSIS METHODS

4.1. Experimental Apparatus. The schematic diagram of the experimental system is shown in Figure 9. The 20 L spherical-explosion-test device complies with the ISO6184-1. It consists of a 20 L stainless steel spherical tank, main control...
computer, gas distribution system, powder release system, data acquisition system, and ignition system. The spectrum acquisition system consists of a grating spectrometer and a spectrum acquisition probe.

The 20 L stainless steel spherical tank with a double-layered structure is designed to withstand a pressure of 2.5 MPa. There is an observation window made of an explosion-proof glass with a compressive strength of 4 MPa at the tank. The process of ignition and explosion in the chamber can be observed from the window. The pressure sensor is a piezoelectric high-sensitivity sensor with a pressure test range of 0−1.7 MPa. The ignition system consists of a high-voltage pulse generator and ignition electricity. The ignition electrodes are located at the center of the tank, and the ignition energy in this experiment was 10 J. The gas distribution was measured according to the law of the Dalton partial pressure method.

The grating spectrometer was produced by Ocean Optics, whose model number is Ocean view HP2000+. As shown in Figure 10, the grating spectrometer consists of a light source, collimating system, dispersion system, and detection system. This spectrometer can cover a wavelength range of 1000 nm for a single shot with a grating resolution of 1 nm, and 400 frames can be recorded within 1 s. In this experiment, the light of explosion flame was dispersed into its component wavelengths and acquired by the spectrometer. The voltage on the integral capacitance is calculated one by one by the measuring system, and the spectral intensity is determined according to the calculations.
4.2. Experimental Method and Conditions. The purity of methane used in this experiment is over 99.0%. The mean diameter of NaHCO₃ particles is 20 μm. The methane concentration in the premixed gas is 10%. The experimental temperature was 16–20 °C, and the humidity of the mixed gas was 40–50% RH. The spectrum acquisition probe is aligned with the observation window to capture the ignition and explosion. A 6 L powder storage tank and a mechanical two-way valve are installed at the bottom of the device to store the preadded powder and spray it into the explosion chamber within 60 ms. When the pressure in the powder storage tank reaches a preset value, the computer defaults to start collecting pressure data and opens the two-way valve. After the powder is sprayed, the valve is closed and the premix gas is ignited after 40 ms. To minimize the impact of accidental factors, the experiment needs to be repeated three times under each condition. In addition, the ignition spectrum data of the igniter is used to remove the influence of the spectrum of the ignition wire during the explosion. The specific experimental scheme is shown in Table 1.

Table 1. Experimental Condition

| concentration of NaHCO₃ (mg/L) | spectral band (nm) |
|-------------------------------|-------------------|
| 0                            | 200–1200          |
| 12.5                         | 200–1200          |
| 25                           | 200–1200          |
| 50                           | 200–1200          |
| 60                           | 200–1200          |

4.3. Analysis Method. 4.3.1. Qualitative Analysis. First, the influence of the ignition spectrum and background spectrum needs to be removed. Then, the sorts of excited radicals present in the flame spectrum are determined by a comparison based on Gaydon’s study. If there is a peak at a characteristic wavelength, it can be believed that a corresponding kind of excited radicals exists. After screening spectrum curves, the number of frames where there is a peak can be determined. Since the frequency of the spectrometer is 400 Hz, the time interval between each single frame Δt is 2.5 ms, so the duration of excited radicals T can be estimated.

\[ T \approx n \times \Delta t \]

4.3.2. Semiquantitative Analysis. According to the weighted factor method, there is a positive correlation between the relative spectral intensity and the concentration of excited radicals. The change of the relative intensity of the spectral intensity with time can reflect the change of the relative content of free radicals/molecules with time. That means the change of the relative spectral intensity with time can reflect the change of the relative content of excited radicals with time. In this way, the influence of the concentration of explosion inhibitors on the content of various excited radicals can be determined.

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Notes

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