Analysis of Test Method of Black Powder by Raman Laser Spectroscopy

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Abstract: Raman laser spectroscopy, as a testing technology that can provide fast, simple, repeatable, and non-damage qualitative and quantitative analysis, is increasingly used in explosion-proof security to detect dangerous goods. Because Raman laser has high energy and some explosives are sensitive to external energy. In this paper, the reaction of black powder to Raman laser under different test conditions is studied, to avoid hazards during testing. The result of research shows that, Raman laser spectroscopic explosive detector can detect black powder only when the laser reaches a certain intensity and integration time. With the increase of laser energy and the increase of integration time, the probability of black powder combustion gradually increases. In the air, black powder can be ignited when the Raman laser intensity reaches 175mW and the integrating time reaches 100ms. Black powder can be detected when the laser intensity reaches 330mW and the integrating time reaches 500ms. When the black powder is placed in water for detection, it is not easy to ignite. When the laser intensity reaches 330mW and the integral reaches 3s, the black powder can be detected. Therefore, when Raman spectrum is used to detect suspected explosive substances, it is not recommended to directly detect unknown objects, but to detect their water suspension, or a combination of various detection methods can be adopted.

Keywords: Raman spectrum; black powder; laser intensity; burning

1. Introduction

As a mature technology, Raman laser spectroscopy has a wide range of applications in many fields. It has the advantages of rich information, no need to pretreatment the object to be measured, no damage to the original material, little interference with water, fast measurement speed and high sensitivity. It has been applied in many industries such as gem identification, food and environmental protection for many years [1-3].

Since the 1980s, Raman laser spectroscopy has been applied to explosive analysis at home and abroad. With the development of technology, at present, portable Raman laser spectrum explosive detector, ion mobility spectrum explosive detector, fluorescent polymer explosive detector, infrared spectrum explosive detector, etc., are several commonly used explosive detectors for security inspection and explosive search and disposal personnel [4].

Judging from all kinds of explosion-related cases and the types of explosives seized by security check, there are various types of explosives used by criminals, and private explosives are relatively common. How to judge and identify private explosives is an important job for security check personnel and explosive search and disposal personnel. However, the components of private explosives are complex, and some components may be particularly sensitive, which are likely to react under the stimulation of high energy Raman laser, and may cause serious consequences such as explosion once stimulated [5-7].This paper takes the common private explosives with high sensitivity including black powder as the research object, analyzes the sensitivity of black powder to Raman laser, and selects the correct detection method, so as to provide reference for security check and explosive search and disposal personnel, and prevent explosion danger.

2. Experimental

2.1. Samples and Instruments

Standard small particle black powder (composition ratio of about 15% toner, 10% sulfur, 75% potassium nitrate) was selected as the sample to be tested.

The SSR-3010 portable Raman laser spectrometer was used in the experiment. The laser wavelength was 785nm and the focus was 7.5mm. The 785nm laser was near infrared. Laser output power is expressed by laser intensity, divided into low (30mW), low (100mW), medium (220mW), high (350mW), high (480mW) five grades. The parameter related to the laser output duration is integral time, which is divided into MS level and S level. Ms level can be adjusted by 100ms step from 100ms to 900ms, and S level can be adjusted by 1s step from 1s to 5s.The spectral range is 150-4300cm^{-1}, which can represent the Raman shift of all substances within this range, that is, the Characteristic peak of Raman can be intuitively represented.

2.2. Experimental Process

In the first step, since the black powder sample is placed in the quartz tube for detection, the actual energy intensity of the Raman laser irradiation on the black powder is measured by the laser energy tester across the wall of the quartz tube.

In the second step, 150mg black powder particles were placed in the transparent quartz glass tube, and the probe of the Raman spectrum detector was vertically pressed against the wall of the quartz glass tube. The test conditions were divided into 14 grades: I×100ms (I=1-9) and 1-5s according to the integral duration. Laser power low (30mW), low medium (100mW), medium (200-250mW), medium high (350mW), high (480mW) five levels of combination, test in turn.

In the third step, 150mg black powder particles were added to the quartz glass tube containing 2ml deionized water. The probe of the Raman spectrum detector was vertically pressed against the wall of the quartz tube. The test conditions were divided into 14 levels: I ×100ms (I =1-9) and 1-5s according to the integral duration. Laser power low (30mW), low medium (100mW), medium (220mW), medium high (350mW), high (480mW) five levels of combination, test in turn.

3. Experimental Results and Analysis

3.1. Measured Results and Analysis of Raman Laser Intensity

The experiment was carried out under the conditions of ambient temperature 25±3℃ and air humidity 55%. There was a layer of quartz tube wall between the laser energy receiver and the Raman spectrum detector probe. The actual laser energy of five levels of laser power, namely low (30mW), low medium (100mW), medium (200-250mW), medium high (350mW) and high (480mW), was tested respectively, and three parallel tests were carried out for each level. The experimental results are shown in Table 1.

Table 1. Measured laser energy of Raman laser probe

Focal length	Laser intensity	Measured value
7.5mm	high	412mW
7.5mm	medium to high	330mW
7.5mm	middle	175mW
7.5mm	lower middle	78mW
7.5mm	low	26mW

3.2. Direct Detection of Black Powder by Raman Laser Spectrometer

Taking 150mg of black powder particles placed in a transparent quartz glass tube, set up the laser intensity and the integral time, Raman spectra detector probe vertical resist from quartz glass tube wall outside, launch the test button, you can see the red laser point focusing on black powder, according to different combinations of laser intensity and integration time, in order to test. The test results are shown in Table 2.

Table 2. Results of direct detection in air

Out put intensity	Integral time	Number of unburned	Number of combustion	Undetected times	Detection times
Low	100ms	3	0	3	0
Low	5s	3	0	3	0
Lower middle	5s	3	0	3	0
Middle	100ms	1	2	3	0
Middle	200ms	0	3	3	0
Medium to high	400ms	0	3	3	0
Medium to high	500ms	0	3	0	3
Medium to high	600ms	0	3	0	3
Medium to high	1s	0	3	0	3

As can be seen from Table 2, when the output power is at or below the medium-low level, no matter how long the integration time is, the black powder does not burn, but the power intensity of Raman laser at this time is insufficient to detect the black powder. When the output power is adjusted to the medium range and the integrating time is set at the minimum of 100ms, combustion may occur during detection. When the output power is at the medium range and the integrating time is ≧ 200ms, combustion phenomenon has definitely occurred, but black powder still cannot be detected. When the output intensity is set to medium and high and the integrating time is set to 400ms, black powder cannot be detected. When the integration time is 500ms, black powder is detected for many times.

3.3. The Results and Analysis of the Detection of Black Powder Water Turbidity by Raman Laser Spectrometer

Taking 150mg of black powder particles placed in a transparent quartz glass tube, and adding suitable amount of water to form black powder fluid, set up the laser intensity and the integral time, Raman spectra detector probe vertical resist from quartz glass tube wall outside, launch the test button, can see the red laser point focusing on black powder, according to different combinations of laser intensity and integration time, Test in turn. The test results are shown in Table 3.

Table 3. Measurement results of turbid liquid in black powder water

Out put intensity	Integral time	Undetected times	Detection times
Middle	5s	3	0
Medium to high	500ms	3	0
Medium to high	1s	3	0
Medium to high	2s	3	0
Medium to high	3s	0	3

It can be seen from Table3 that when the output intensity is set to medium or below, no matter how long the integration time is, the detection of mixed water cannot be detected. When the output intensity is further improved to the medium and high grade, the integration time is gradually extended, and the detection can be reliably detected when it reaches 3s.

Both of them require medium and high laser intensity, but the integral time is increased from 500ms to 3s, indicating that water has an obvious absorption effect on the energy intensity of Raman laser, which significantly reduces the energy intensity irradiated to the surface of black powder.

4. Discussion of Results

As a relatively mature method of detection, Raman laser spectroscopy has been widely used in many fields. In the field of explosion-proof security check, the safety of Raman laser spectrum explosive detector has always been the focus of attention in the industry due to the particularity of explosives and explosives. This paper systematically studied the testing effect and phenomenon of Raman laser spectrum in the process of black powder detection, and the research conclusions are as follows:

(1) When the black powder is directly detected by Raman laser spectroscopy explosive detector in air, it is found that when the Raman laser intensity is low and medium low (≦78mW), the black powder will not be burned by Raman laser, but the components of black powder can not be detected by this Raman laser intensity; When the intensity of Raman laser reaches to medium (175mW), the Raman laser has a high probability of causing the combustion of black powder, and the composition of black powder can not be detected under this laser intensity. When the strength of Raman laser reaches to medium and high (330mW), the Raman laser will cause the combustion of black powder. When the integration time is 400ms, the components of black powder cannot be detected; when the integration time is ≧500ms, the components of black powder can be detected.

(2) It is found that when the intensity of Raman laser is low, medium low and medium (≦ 175mW), it does not cause the combustion of black powder, but it can not detect the composition of black powder. When the intensity of Raman laser reaches medium and high (330mW) and the integration time is less than 3s, the composition of black powder cannot be measured; when the integration time is ≧3s, the composition of black powder can be measured safely.

(3) It can be seen from the results of the direct detection of black powder and the detection of black powder water and turbidity liquid that the water can absorb part of the Raman laser energy and reduce the laser intensity on the surface of black powder.

(4) In the process of explosion proof security check, when the unknown explosive dangerous goods are tested, the Raman laser spectrum explosive detector cannot be used for direct detection. It is suggested to make the samples into turbidity liquid for detection, or to use the Raman laser spectrum explosive detector with other explosive detectors.

(5) It is necessary to make explosive dangerous goods into turbidized liquid for detection, so that the advantages of Raman laser spectrodynamite detector, compared with other spectrometers, can not be reflected without pretreatment of the substances to be measured.

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