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# Investigation on Stripping-down TNT from Waste Munitions by Supercritical $CO_2$ Fluid Extraction under Low Temperature Conditions

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**Abstract:** The traditional methods of waste munitions treatment are expensive and also have potential risks during the treatment process. The supercritical fluid extraction technique has been a rapidly developing technique in the chemical industry in recent years. $CO_2$ is used as the solvent, which has some advantages, such as low pollution, low cost, good chemical stability and can be operated under low temperature conditions. This research explored the feasibility of reclaiming TNT from waste munitions by supercritical $CO_2$ fluid extraction. It was found interestingly that the melting point of TNT can be lowered in supercritical $CO_2$ fluid. Therefore, the melting process of TNT was observed under different temperature and pressure conditions to determine the best operating conditions for stripping-down TNT from waste munitions. Afterwards, simulated warheads with weight loadings of 60 g, 500 g and 1 kg of TNT were prepared and stripping-down tests from the simulated warheads were carried out using supercritical $CO_2$ fluid at temperatures lower than the normal melting point of TNT. The results showed that TNT could be completely removed from the simulated warheads and the optimum operating conditions were determined as 55 °C and 25 MPa. This study will contribute to the feasibility evaluation of stripping-down TNT-based high explosives.

**Keywords:** treatment of waste munitions, supercritical fluid extraction technique, TNT, stripping-down, low temperature conditions

**Supplementary material (S.I.)** available at: http://www.wydawnictwa.ipo.waw.pl/CEJEM/contents/2018/vol-15-no-1.html

## 1 Introduction

The disposal of munitions and explosives over the last hundred years has led to some serious detonation events and widespread contamination of the environment. Current methods for the disposal of munitions containing melt-cast explosives (TNT, Composition B, and Tritonal) include open burning/open detonation (OB/OD), steam out, wash out and contour drilling. The environmental impacts associated with these methods include air and ground contamination and waste stream treatment. Furthermore, these disposal methods are often wasteful, resulting in the destruction of potentially valuable energetic materials. Therefore, alternative, environmentally-friendly technologies need to be developed. The resource recovery of energetic materials should also be an important consideration in the development of new disposal technologies [1-3].

Trinitrotoluene (TNT) is one of the most commonly used explosives for military applications because of its low melting point and its resistance to shock or friction, which allow it to be handled, stored, and used with comparative safety [4]. Many military high explosive formulations based on TNT are used in a variety of military ordnance applications, such as bursting charges for projectile warheads and land mines. But the disposal and recovery of munitions containing TNT is a really troublesome problem. Typical methods for extracting TNT from the high explosives are melt-out techniques, which can be divided into two categories, those which use pressurized hot water or steam to melt the filling and those which use indirect methods. The most important indirect methods are microwave and induction melt-out [1, 5]. These methods are disadvantageous for being time consuming, inappropriate for mass reclamation of TNT, generating too much polluted waste-water, which is required to be treated at prohibitively high cost, and being of low efficiency.

The supercritical fluid extraction technique using carbon dioxide ($CO_2$) has been recognized as a green technology, which has also been a rapidly developing technique for the chemical industry in recent years [6, 7]. $CO_2$ is a clean and versatile solvent with gas-like diffusivity and liquid-like density in the supercritical phase, which has provided an excellent alternative to the use of chemical solvents. In particular, it has significant potential in a specific and very important area of research associated with the manufacture, processing and destruction of high energy materials (HEMs) [8-13]. The disposal of munitions and explosives is extremely risky to personnel, and any improvements that would reduce the fire and explosion risks of the disposal processes are highly desirable [14-16]. Considering the factors of industrial safety and environmental protection, the supercritical fluid extraction technique may be a better method for the disposal and recovery of munitions containing TNT. Teipel *et al.* [17] reported an extensive set of data on the solubility of TNT in supercritical $CO_2$ fluid (SC $CO_2$). These solubility measurements cover a pressure range of 15 MPa to 50 MPa and a temperature range of 303 K to 413 K. At the highest combinations of temperature and pressure, the experimental data indicate that the solubility of TNT is about 55 mg/g $CO_2$. Morris *et al.* [18-20] have presented a method for the extraction of TNT from high explosives using SC $CO_2$ fluid. This method recovers TNT at a temperature above the melting point (*e.g.* 85 °C) of TNT. However, it is known that there may be other explosive components in the high explosive. Therefore, it is very possible that unexpected explosions may occur during the extraction process [21]. This is very dangerous. Furthermore, TNT has a very low solubility in SC $CO_2$ fluid. Therefore, a large volume of SC $CO_2$ fluid is required for recovering TNT from high explosives, and this significantly increases the cost of the treatment.

There is a particularly noteworthy phenomenon that often occurs during the supercritical fluid extraction/separation processes. The solid can be melted at temperatures lower than its normal melting point in the presence of a supercritical fluid [22-24]. Lucien and Foster [22] have pointed out that the melting point of a pure solid can be depressed significantly under the influence of high pressure $CO_2$ and a similar phenomenon exists in the case of the eutectic points of solid mixtures. The formation of a liquid phase under high pressure may or may not be accompanied with excess solid so that both solid-liquid-vapour (S-L-V) and liquid-vapour (L-V) equilibria are possible. Lian *et al.* [23] have developed a method based on the Clapeyron equation for two-component, three-phase equilibria, for predicting the maximum melting point depression of a compound in the presence of a supercritical fluid. Pasquali *et al.* [24] have demonstrated that the melting temperature of some polymers decreases linearly with increasing pressure at low pressures, and is followed by an approximately constant region at higher pressures. Our research group [25] has developed a method for melting-out TNT from high explosive warheads using SC $CO_2$ fluid extraction, but this study did not touch upon the melting point depression phenomenon of TNT in SC $CO_2$ fluid due to the constraints of the experimental apparatus.

In the present study, the melting point depression phenomenon of TNT in SC $CO_2$ fluid was observed by means of supercritical fluid equipment under different temperature and pressure conditions. The optimal operating conditions were also determined for stripping-down TNT from waste munitions by SC $CO_2$ fluid extraction.

Afterwards simulated warheads with weight loadings of 60 g, 500 g and 1 kg of TNT were prepared and stripping-down tests of TNT from the simulated warheads were carried out using SC $CO_2$ fluid to identify the optimal operating conditions. It is anticipated that these research results will be useful for solving the problems of the disposal of waste munitions for industrial safety and environmental protection. In addition, the waste explosives can also be recovered for re-use.

# 2 Experimental

## 2.1 Materials

Technical grade TNT (2,4,6-trinitrotoluene) with a purity of 99.9% was obtained from the 205$^{th}$ Arsenal in Taiwan, which has actually been used in a variety of military applications.

## 2.2 Preparation of test samples

The TNT was melted in a hot water bath at 90 °C and hand-poured into empty steel shells to create cylindrical pellets. The length and diameter of each cylindrical pellet were 30.0 ±0.1 mm and 10.0 ±0.1 mm, respectively. The densities of these pellets were ~1.53 ±0.03 g/cm$^3$. Afterwards, a cylindrical pellet was fixed on the bottom of a glass sample bottle as shown in Figure 1, in order to observe the melting point depression phenomenon of TNT in SC $CO_2$ fluid. Furthermore, about 60 g, 500 g and 1 kg of pure TNT were melt-loaded into simulated warheads for the stripping-down tests of TNT from the simulated warheads as shown in Figures 2-4.

**Figure 1.** Cylindrical TNT pellet and glass sample bottle

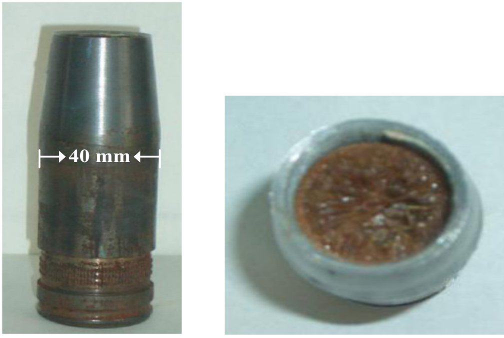



**Figure 2.** Simulated warhead with a weight loading of 60 g of TNT

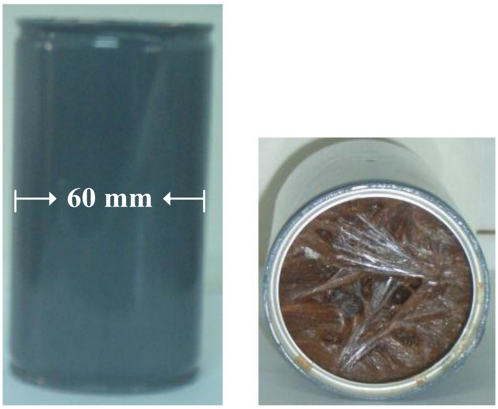



**Figure 3.** Simulated warhead with a weight loading of 500 g of TNT

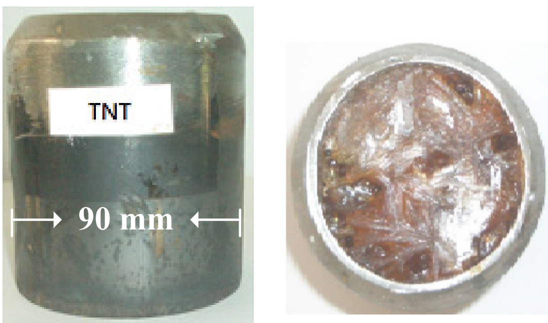



**Figure 4.** Simulated warhead with a weight loading of 1 kg of TNT

## 2.3 Apparatus and procedures

Pilot-scale supercritical fluid extraction equipment with a viewing window, which was designed and made by Taiwan Supercritical Technology Co., Ltd. as shown in Figure 5, was used to observe the melting process of TNT in SC $CO_2$ fluid. This equipment had a 350 mL pressure vessel and the maximum operating pressure and temperature were 60 MPa and 70 °C, respectively. The melting point depression phenomenon of TNT in SC $CO_2$ fluid was experimentally explored over the temperature and pressure ranges of 25-65 °C and 5-50 MPa, respectively. The residence time of all test samples in the pressure vessel was set at 50 min. There were 35 preset testing conditions as shown in Table 1. The experimental results were used to evaluate the appropriate testing conditions for stripping-down TNT from the simulated warheads.

**Figure 5.** Pilot-scale supercritical fluid extraction equipment with a viewing window

**Table 1.** Experimental conditions for testing melting point depression

| Test No. | Temperature [°C] | Pressure [MPa] | Number of tests |
|---|---|---|---|
| 1-7 | 25 | 5, 10, 15, 20, 25, 30, 50 | 7 |
| 8-14 | 35 | 5, 10, 15, 20, 25, 30, 50 | 7 |
| 15-21 | 45 | 5, 10, 15, 20, 25, 30, 50 | 7 |
| 22-28 | 55 | 5, 10, 15, 20, 25, 30, 50 | 7 |
| 29-35 | 65 | 5, 10, 15, 20, 25, 30, 50 | 7 |

A further experimental apparatus for the investigation of the stripping-down of TNT from the simulated warheads was SC-2000 supercritical fluid extraction equipment from Taiwan Supercritical Technology Co., Ltd. as shown in Figure 6, which included a 2-L pressure vessel and a 2-L recovery vessel. The maximum operating pressure and temperature were 70 MPa and 80 °C, respectively. Simulated warheads with a weight loading of 60 g of TNT were used to determine the optimal operating condition for stripping-down TNT from the warhead over the temperature, pressure and residence time ranges of 35-65 °C, 15-35 MPa and 5-30 min, respectively. Finally, the optimal operating conditions were used to verify the removal of TNT from simulated warheads with weight loadings of 500 g and 1 kg of TNT. The collection of the recovered TNT was in two parts. The main part was the melting-out of TNT from the simulated warhead, and the another part was the dissolving-out of TNT by SC $CO_2$ fluid. In the experimental process, the 'melting-out' TNT was collected in the pressure vessel and the 'dissolving-out' TNT was collected in the recovery vessel. However, the recovered amount of 'dissolving-out' TNT should be very low, because TNT has a very low solubility in SC $CO_2$ fluid. The amount of TNT removed from the simulated warhead is close to the amount of 'melting-out' TNT from the simulated warhead.

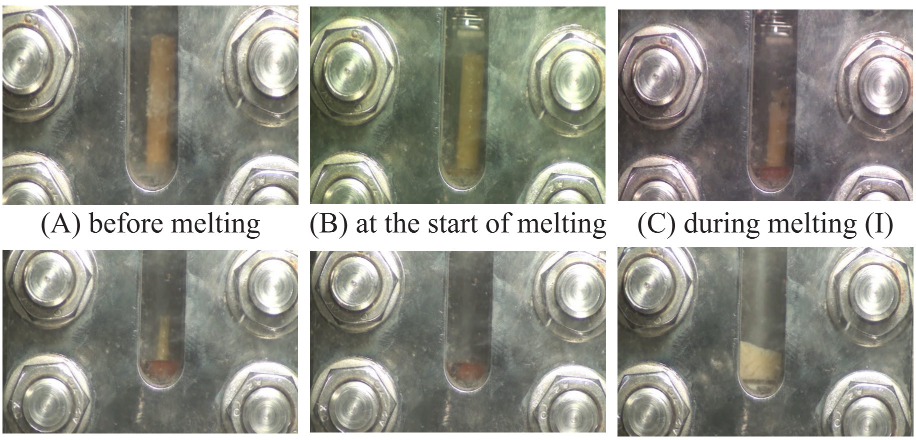
**Figure 6.** SC-2000 supercritical fluid extraction equipment

# 3 Results and Discussion

## 3.1 Observation of melting point depression of TNT in SC-$CO_2$

This study observed the morphological change of cylindrical TNT pellets before and after the experiment to determine whether TNT exhibited a phase change in SC $CO_2$ fluid under various temperature and pressure conditions. Figure 7 presents photographs showing the results from *in situ* observations of the melting process of the cylindrical TNT pellets during the experimental period.

Figure 7(A) shows the morphology of the cylindrical TNT pellet before melting. The cylindrical TNT pellet was fixed to the bottom of the glass sample bottle and placed in the apparatus. The operating pressure and temperature were set at 10 MPa and 55 °C, respectively. Figure 7(B) illustrates that the solid phase started to melt at 55 °C, which is lower than the theoretical melting point of TNT at 25 °C and 1 atm (about 80 °C). Figures 7(C) and 7(D) show the morphological changes of the cylindrical TNT pellet during melting. The melting TNT dropped to the bottom of the glass sample bottle, and the height of the cylindrical TNT pellet gradually decreased. The end of the melting process of TNT is shown in Figure 7(E). When the operating pressure was released (returned to 1 atm) and the SC $CO_2$ was quickly removed, there was some crystalline TNT present in the glass sample bottle because some TNT dissolved in the SC $CO_2$ fluid and was precipitated as shown in Figure 7(F). The total amount of TNT in the glass sample bottle was reduced because a little TNT was dissolved in the SC $CO_2$ fluid and removed with it.



(A) before melting (B) at the start of melting (C) during melting (I)

(D) during melting (II) (E) at the end of melting (F) after pressure release

**Figure 7.** Photographs showing results from *in situ* observations of the melting process of a cylindrical TNT pellet during the experimental period

Figure 8 shows the morphological changes of cylindrical TNT pellets after experiments at a constant temperature of 25 °C and pressure range of 5 MPA to 50 MPa. The up and down, two rows of photographs are the front views and rear views, respectively. It was observed that the melting phenomenon had not occurred. However, there was some recrystallized TNT on the bottom of the glass sample bottle because the TNT dissolved in the $CO_2$ fluid was precipitated after fluid expansion. The recrystallized TNT forms loose needle-shaped aggregates.

Figure S1 in S.I. shows the morphological changes of the cylindrical TNT pellets at a constant temperature of 35 °C and pressure range of 5 MPa to 50 MPa. The melting phenomenon was also not observed at pressures of 5 MPa, 10 MPa and 15 MPa. There was only a small amount of recrystallized TNT on the bottom of the glass sample bottle. However, the melting phenomenon was observed in the pressure range of 20-50 MPa. It was observed that the TNT accumulated at the bottom of the bottle solidified with a deeper colour when the experimental pressure was returned to atmospheric pressure. Figures S2-S4 in S.I. show the experimental results at constant temperatures of 45 °C, 55 °C and 65 °C, respectively, and the melting phenomenon was observed in the pressure ranges of 15-50 MPa, 10-50 MPa and 10-50 MPa, respectively. The normal melting point of TNT at 1 atmosphere pressure is close to 80 °C. These experimental results indicated that TNT can be melted at lower temperatures (45 °C, 55 °C and 65 °C) in SC $CO_2$ fluid. The melting conditions of TNT in SC $CO_2$ fluid are marked on the $CO_2$ phase diagram shown in Figure 9. In addition, it is also observed that the melting point of TNT has a downward trend when the pressure of the SC $CO_2$ fluid is increased.

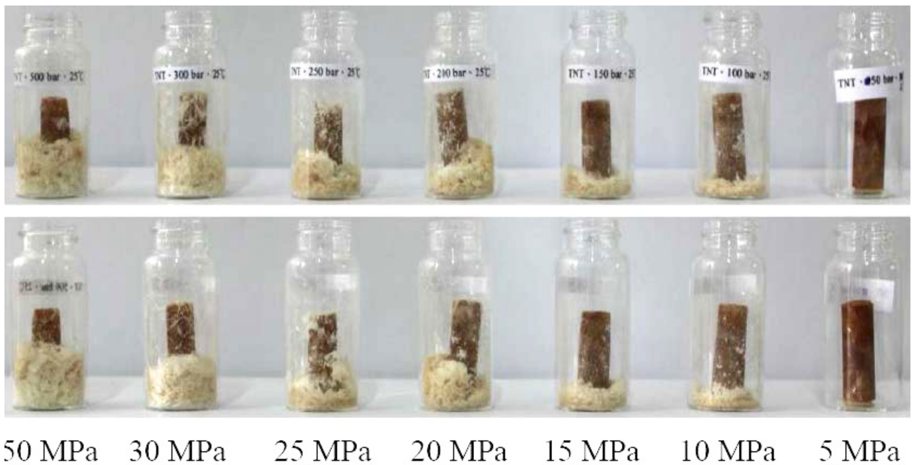



**Figure 8.** Morphological changes of cylindrical TNT pellets after the experiments at a constant temperature of 25 °C and pressure ranging from 5 MPa to 50 MPa

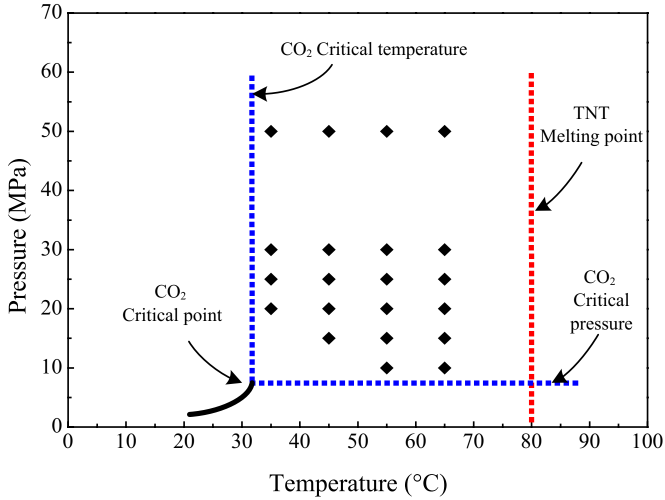



**Figure 9.** Melting conditions of TNT in supercritical $CO_2$ fluid

## 3.2 Determination of the optimal operating conditions for stripping-down TNT from warheads

Considering the factors of improving industrial safety and reducing energy consumption, the lower the operating pressure and temperature are, the better are the operating conditions in an industrial process for stripping-down TNT from waste munitions.

Simulated warheads with a weight loading of 60 g of TNT were used to determine the optimal operating conditions for stripping-down TNT from a warhead using SC $CO_2$ fluid extraction. Initially, the experimental conditions were set at a constant pressure of 25 MPa and temperature range of 35 °C to 65 °C. The residence time in the pressure vessel in all of the experiments was set at 30 min, in order to measure the removal efficiency of TNT from the simulated warhead. The experimental results indicated that the removal efficiency of TNT increased with increasing operating temperature, as shown in Table 2. The removal of TNT can reach 100% when the operating temperature is higher than 50 °C. Furthermore, the removal efficiency of TNT at residence times of 5 min, 10 min and 20 min were examined under conditions where the operating temperatures were 50 °C and 55 °C, respectively. These experimental results are also shown in Table 2. Figure 10 shows the removal efficiencies of TNT at residence times of 5 min, 10 min and 20 min under the operating pressure and temperature conditions of 25 MPa and 55 °C. It was found that the removal efficiency of TNT can reach 100% within 10 min residence time.

**Figure 10.** Removal efficiencies of TNT for the simulated warhead with a weight loading of 60 g of TNT at residence times of 5 min, 10 min and 20 min under the operating pressure and temperature conditions of 25 MPa and 55 °C

**Table 2.** Removal efficiency of TNT at a constant pressure of 25 MPa and various temperature and residence time conditions

| Operating pressure [MPa] | Operating temperature [°C] | Residence time [min] | Removal efficiency of TNT [%] |
|---|---|---|---|
| 25 | 65 | 30 | 100 |
| | 55 | 30 | 100 |
| | | 20 | 100 |
| | | 10 | 100 |
| | | 5 | 18.8 |
| | 50 | 30 | 100 |
| | | 20 | 100 |
| | | 10 | 17.3 |
| | | 5 | 1.8 |
| | 45 | 30 | 6.1 |
| | 35 | 30 | 1.2 |

In order to determine the optimal operating conditions for stripping-down TNT from a warhead, the experimental conditions were also set at a constant temperature of 55 °C and a pressure range of 15 MPa to 35 MPa. The residence time of all experiments was also set at 30 min. The experimental results indicated that the removal efficiency of TNT increases with increasing operating pressure as shown in Table 3. Furthermore, the removal efficiences of TNT at residence times of 5 min, 10 min and 20 min were examined under operating pressure of 20 MPa and 25 MPa, respectively. The experimental results are also listed in Table 3. The best experimental result was that the removal efficiency of TNT can reach 100% within 10 min residence time under the operating conditions of 55 °C and 25 MPa. Therefore, it was experimentally confirmed that TNT can be completely removed from the simulated warhead at temperatures much lower than its normal melting point by SC $CO_2$ fluid extraction. In a practical industrial application, the optimal operating conditions may be located at a temperature of 55 °C and a pressure of 25 MPa.

**Table 3.** Removal efficiency of TNT at a constant temperature of 55 °C and various pressure and residence time conditions

| Operating temperature [°C] | Operating pressure [MPa] | Residence time [min] | Removal efficiency of TNT [%] |
|---|---|---|---|
| 55 | 35 | 30 | 100 |
| | 30 | 30 | 100 |
| | 25 | 30 | 100 |
| | | 20 | 100 |
| | | 10 | 100 |
| | | 5 | 18.8 |
| | 20 | 30 | 100 |
| | | 20 | 100 |
| | | 10 | 17.3 |
| | | 5 | 2.3 |
| | 15 | 30 | 64.6 |

### 3.3 Feasibility study for industrial scale-up

Simulated warheads with weight loadings of 500 g and 1 kg of TNT were used to evaluate the feasibility of industrial scale-up for stripping-down TNT from warheads by SC $CO_2$ fluid extraction under low temperature conditions. The operating pressure, temperature and residence time were set at 25 MPa, 55 °C, and 30 min, respectively. Figure 11 shows the removal efficiency of TNT for the simulated warhead with a weight loading of 500 g of TNT under the set operating conditions. It was found that TNT can be melted and completely removed from the simulated warhead by SC $CO_2$ fluid extraction, and then drops into a glass beaker. The removed TNT is solidified and collected completely in a glass beaker, as shown in Figure 11(c). The simulated warhead with a weight loading of 1 kg of TNT was also tested under the same operating conditions. The experimental result is shown in (S.I.) Figure S5. Again, TNT was also completely removed from the simulated warhead. Therefore, the feasibility of industrial scale-up for stripping-down TNT from waste munitions by SC $CO_2$

fluid extraction was successfully demonstrated through these two experiments. However, the practical operating conditions must still be tested and evaluated using industrial grade apparatus in industrial applications.

(a) 5 min (b) 10 min (c) 20 min

**Figure 11.** Removal efficiency of TNT for the simulated warhead with a weight loading of 500 g of TNT at a residence time of 30 min under the operating pressure and temperature conditions of 25 MPa and 55 °C

## 4 Conclusions

The feasibility of the industrial application of stripping-down TNT from waste munitions using SC $CO_2$ fluid extraction under low temperature conditions was successfully demonstrated in this study. From the above experiments and analyses, the following conclusions were obtained:

(1) TNT can be melted at temperatures lower than its normal melting point in the presence of SC $CO_2$ fluid. The experimental results indicated that the melting point of TNT exhibits a downward trend when the pressure of SC $CO_2$ fluid is increased.

(2) It was experimentally confirmed that TNT can be removed completely from simulated warheads by SC $CO_2$ fluid extraction. The removal efficiency of the TNT increases with increasing operating pressure, temperature and residence time.

(3) The feasibility of industrial scale-up was experimentally demonstrated. In a practical industrial application, the best operating pressure, temperature and residence time can be set to 25 MPa, 55 °C, and 30 min, respectively.

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