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# Effect of microwave irradiation on TATB explosive

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ABSTRACT

Finished TATB (1,3,5-triamino-2,4,6-trinitrobenzene) explosive safety under 800 W microwave irradiation was experimented. No burning, deflagration and detonation were observed during 30-min continuous irradiation and no remarkable change were observed after irradiation according to HPLC, particles size analysis, and differential thermal analysis. Wet TATB sampled from synthesis line was irradiated with microwave vacuum method and irradiated TATB was measured to accord with military standard specifications including appearance, moisture and volatile, chloride content, HPLC, mean particle size, DTA exothermic peak, ash, acetone soluble content, PH value, etc. Microwave vacuum desiccation was deemed laborsaving, energy-efficient, and practicable compared to conventional processing method.


## 1. Introduction

Explosives were experimented not to burn, deflagrate, or detonate under low and high power microwave irradiation [1,2] and could be melted and recovered safely from inside obsolete huge bombs with microwave heating method [3]. However susceptive impurities would form overheating spot and make explosives mixture more hazardous to microwave irradiation [4–6]. Furthermore explosives with smaller particle size would be slightly more susceptive of microwave induced dielectric breakdown [7].

TATB (1,3,5-triamino-2,4,6-trinitrobenzene), an extremely insensitive high explosives (IHEs) [8], was reviewed in our lab and its impurities seemed a little bothersome in preliminary experiments [9]. Here we showed a safe irradiating operation and their feasibility as drying method.

## 2. Experimental

### 2.1. Reagents and instrument

*Reagent*: Finished TATB, finished product, made from wet-amination method [8] in our laboratory; wet TATB, sampled from synthesis line in our lab, after washing and filtering, before drying.

*Instrument*: Microwave oven (tailor-made, 2.45 GHz, 800 W, remote control); particle size analyzer (LS230, from Beckman Coulter Inc.); Differential Thermal Analyzer (WCT-1, from Beijing optic instrument factory); HPLC (Agilent 220, from Agilent Inc.).

### 2.2. Experiments

Finished TATB was irradiated in microwave oven programmable and interruptable in case of emergency, which was processed inside explosion-proof chamber with remote control to avoid damage by security issues. No damages were observed during irradiation. Wet TATB was then irradiated within that apparatus supplemented with vacuum pipeline. Comparison of origin and irradiated samples were carried out according to items mentioned in military specification [10].

## 3. Results and discussion

### 3.1. Irradiation of finished TATB

Irradiation of finished TATB was processed gently from mildest parameters including quantity 0.05 g, period 5 s, and power 160 W to oven scale parameters including quantity 200 g, period 20 min continuously, and power 800 W. No deflagration or detonation was observed with their appearance unchanged. It is confirmed that finished TATB as pure explosive should be safe under microwave irradiation.

It sounded inconceivable that so much irradiation could not initiate TATB. It was suggested that entirely symmetrical TATB molecules absorb irradiation really difficultly, major irradiation was

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**Table 1**
Particles size of origin and irradiated TATB samples.

| Sample | Microwave power (W) | Irradiation duration (min) | Mean particles size (μm) | Middle particles size (μm) | Peak particles size (μm) | Surface area ($cm^2/g$) |
|---|---|---|---|---|---|---|
| TATB-45-1 | 800 | 0 | 13.65 | 13.75 | 18.00 | 9,633 |
| TATB-45-2 | 800 | 5 | 13.65 | 13.81 | 18.00 | 10,773 |
| TATB-45-3 | 800 | 10 | 13.55 | 13.71 | 18.00 | 11,452 |
| TATB-45-4 | 800 | 15 | 13.39 | 13.64 | 18.00 | 10,982 |
| TATB-45-5 | 800 | 20 | 13.30 | 13.46 | 18.00 | 10,621 |

absorbed by samples, wall, and atmosphere inside oven equably, and heat dissipation also works. Microwave irradiation has not been showed to initiate pure explosives till now.

### 3.2. Finished TATB analysis

Finished TATB and its irradiated samples were compared here. HPLC results with ultraviolet detector ranged from 190 to 900 nm did not reveal evidential decrease of TATB or emergence of new compounds.

As given in Table 1, particle size of samples changed a little after irradiation while their surface area increased about 10.5–18.9% which was notable giving that error of surface area calculated from particle size distribution curve was around 10%. Microwave energy was not considerable to reunite or re-disperse energetic particle [4]. It was supposed here that microwave should be some more powerful than conventional way to excite and dehydrate solvents residue which adhered two small particles to one big particle. It was not sure yet how microwave works in nature.

Differential thermal analyzer data of irradiated samples showed no remarkable change and all accord with specification [10] as given in Table 2.

The aforementioned experiments indicated that pure TATB showed no remarkable change physically and chemically under microwave irradiation. Then what happened to others? Did wet TATB hydrolyze or desiccate during microwave irradiation?

### 3.3. Irradiation trial of wet TATB and Introduction of microwave vacuum method

Our sequential experiments showed that microwave irradiation should not initiate coarse, superfine and submicron TATB but wet TATB emerged once yellow smog and particles surface turn black during irradiation [9]. Volatile components was deemed responsible which released rapidly and departed slowly such as thermolysis substance of ammonium chloride during microwave heating process as given in Table 3 [11,12]. It is reasonable not to refrain or decelerate the process but to accelerate diffusing and departing of gaseous compound so that microwave vacuum method came into being naturally.

**Table 2**
DTA exothermic peak of irradiated TATB samples.

| Sample | Irradiation parameter | Peak value (°C) | Onset value (°C) |
|---|---|---|---|
| TATB-15-1 | – | 393.2 | 377.9 |
| TATB-15-2 | 800 W, 5 min | 395.3 | 379.0 |
| TATB-15-3 | 800 W, 10 min | 393.5 | 378.2 |
| TATB-15-4 | 800 W, 15 min | 392.9 | 379.8 |
| TATB-15-5 | 800 W, 20 min | 392.5 | 378.2 |
| TATB-45-1 | – | 401.5 | 387.5 |
| TATB-45-2 | 800 W, 5 min | 399.1 | 384.0 |
| TATB-45-3 | 800 W, 10 min | 397.2 | 386.0 |
| TATB-45-4 | 800 W, 15 min | 401.1 | 387.1 |
| TATB-45-5 | 800 W, 20 min | 399.9 | 387.9 |
| Preferred value [10] | – | ≥375 | – |

**Table 3**
Components in wet TATB.

| Type | Component | Contents |
|---|---|---|
| Main compound | TATB (1,3,5-triamino-2,4,6-trinitrobenzene) | 50–70% |
| Residual solvent | Water, acetone, toluene | 15–25% |
| Inorganic by-product | Ammonium chloride | ~1% |
| Organic impurity | Poly-nitro-amino-chloro-benzene | 1–3% |

**Table 4**
Processing of wet TATB.

| Batch number | Division and process | | |
|---|---|---|---|
| | Sample 1 | Sample 2 | Sample 3 |
| Batch 1 (300 g) | REF-1 | Microwave 10 min COM-1 | Microwave 24 min MWI-1 |
| Batch 2 (300 g) | REF-2 | Microwave 20 min COM-2 | Microwave 20 min MWI-2 |
| Batch 3 (300 g) | REF-3 | Microwave 30 min COM-3 | Microwave 22 min MWI-3 |

The trial apparatus consisted of drilled oven, sample flask, vacuum pump, explosion proof wall, remote controller, etc. as given in Fig. 1, with which no yellow smog emerged any longer and explosives dust must be separated from circuit region.

### 3.4. Act of wet TATB during microwave vacuum process

As described in Table 4, every batch of three wet TATB samples was divided into three equivalent samples. Sample 1 was heated inside 80 °C oven for 8 h and inside 180 °C oven for another 8 h in turn, same to traditional operation, named reference sample. Sam-

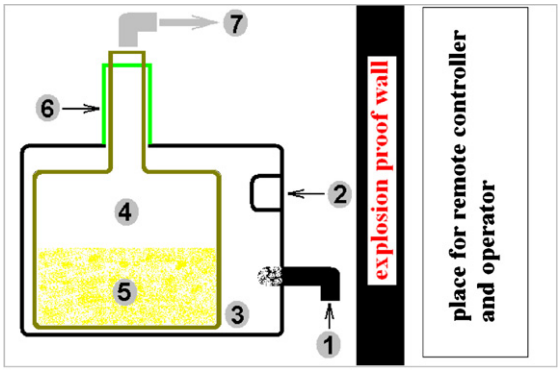


**Fig. 1.** Sketch of microwave vacuum trial apparatus: 1, cable to remote controller; 2, microwave feed-in; 3, microwave irradiation region; 4, vacuum region; 5, TATB sample; 6, brass bushing; 7, to vacuum pump.

**Table 5**
Desiccation results of TATB samples.

| Sample | Appearance | Moisture and volatile, %(m/m) | Chloride content, %(m/m) | HPLC purity, % | Mean particle size, μm |
|---|---|---|---|---|---|
| REF-1 | Light yellow powder | 0.03 | 0.27 | 99.3 | 15.8 |
| MWI-1 | Light yellow powder | 0.06 | 0.29 | 97.8 | 15.3 |
| COM-1 | Light yellow powder | 0.06 | 0.30 | 97.7 | 15.8 |
| REF-2 | Light yellow powder | 0.03 | 0.24 | 97.9 | 15.1 |
| MWI-2 | Light yellow powder | 0.06 | 0.26 | 98.1 | 14.9 |
| COM-2 | Light yellow powder | 0.06 | 0.26 | 98.2 | 15.4 |
| REF-3 | Light yellow powder | 0.03 | 0.27 | 97.5 | 14.1 |
| MWI-3 | Light yellow powder | 0.06 | 0.30 | 98.2 | 15.2 |
| COM-3 | Light yellow powder | 0.06 | 0.29 | 98.3 | 15.1 |
| Preferred value | Light yellow powder | ≤0.10 | ≤0.40 | N/A | N/A |

ple 2 was heated inside 80 °C oven for 8 h and irradiated inside microwave for certain minutes in turn, named comparison sample. Sample 3 was directly irradiated inside 800 W microwave oven till constant weight, named microwave sample. All the nine dried samples were analyzed and their results were shown in Table 5.

All nine samples appeared light yellow powder without macroscopic impurities and should be undistinguishable due to their appearance. Their moisture and volatile contents and chloride contents were measured up to preferred value as given in table.

Their HPLC purity valued 97.5–99.3%, which meant undistinguishable from each other considering analysis error was about ±1.0%.

Mean particles size of samples valued 14.1–15.8 μm, which should be undistinguishable during their application.

Some other items mentioned in military specifications [10] including DTA exothermic peak temperature, ash content, acetone soluble content, and PH value were also measured up. All the data inspiringly showed that three methods are replaceable with each other.

### 3.5. Practicability

Wet TATB should be heated conventionally for 16 h in order to drive away their moisture and inorganic impurity, which could be replaced by microwave vacuum irradiation less than half an hour now. Together with decrease of process duration, energy consumption of microwave would be equal to third—sixth of conventional methods. First, microwave induced heating of polar substance inside samples and heat dissipation to circumstance were relatively less, while conventional method caused heat dissipation really considerable. Secondly, conventional "surface hot and inner cold" manner would cause hardening of surface layer and prohibiting of moisture dissipation from inner to surface while microwave heated surface and inner moisture simultaneously and decreased resistance of moisture dissipation. Thirdly, vacuum here speed volatilization of moisture and promoted drying efficiency compared to conventional method.

## 4. Conclusions

Microwave irradiation did not initiate burning, deflagration and detonation of finished TATB explosives. Irradiated TATB showed no remarkable change due to HPLC measurement, particles size analysis, and differential thermal analysis.

Wet TATB explosives were irradiated with self-fabricated microwave vacuum apparatus. Irradiated samples showed no remarkable difference from comparison samples and reference samples due to measurements results of appearance, moisture and volatile contents, chloride contents, HPLC purity, mean particle size, DTA exothermic peak temperature, ash contents, acetone soluble content, PH value, etc., which indicated that microwave vacuum method should be replaceable of conventional processing method. Microwave vacuum desiccation method should be energy-efficient, timesaving, and practicable.

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