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Rapid Prediction of Effect of Ferrous Iron on the Stability of Emulsion Explosives Matrix

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Abstract: *Designing an emulsified matrix formulation in the laboratory, the ratios of the emulsifier span-80 and T-152 were 1:0, 2:1, 1:2, 0:1 respectively. Then the preparation was added by 0.04% Fe^{2+} impurities emulsion matrix. Based on the water-soluble test method, we used ultrasonic instrument to focus on emulsion matrix containing Fe^{2+} impurities. The results show that the crystallization amount of emulsion matrix by adding Fe^{2+} is more than that of not adding Fe^{2+} .Impurities Fe^{2+} has damaging effects on the stability of its interface membrane. At the same time, ultrasound can accelerate the speed of demulsification of emulsion matrix. As composite emulsifiers span-80: T-152=1:2, the stability of emulsion matrix is the best.*

Keywords: *ultrasonic; emulsion explosive matrix; ferrous ion; stability*

1. Introduction

The stability of emulsion explosive refers to the property that its physical and chemical properties remain unchanged and the detonation performance does not change obviously. It is an important technical index to measure the quality of emulsion explosive[1]. The aqueous phase of emulsion explosive contains a large number of inorganic salts, among which the content of nitric acid is more than 70%. In the production and transportation of nitric acid, there is a certain corrosion of metal containers and pipelines, especially in the production of emulsion explosives, the nitric acid temperature reaches more than 110℃. Metal Fe^{2+} impurities will inevitably enter into the raw materials of emulsion explosives, which has a certain impact on the stability of emulsion explosives[2].

The existing methods for evaluating the stability of emulsion explosive, such as electrical conductivity method and dynamic equilibrium method, can only reflect the stability of emulsion explosive laterally, while the water-soluble method has a common fault with the above methods, that is, the test time is too long. In this experiment, on the basis of water solution method, the interfacial film was destroyed by ultrasonic force, and the nitric acid was accelerated to break free from the oil film by particles, so as to quickly evaluate the stability of emulsified matrix [3].

Reference [4] focuses on the influence of a single emulsified matrix in various test conditions under ultrasonic effect, and reference [5] focuses on the stability difference of emulsified matrix formulated in the laboratory under ultrasonic effect, but the experimental samples are quite different from the current emulsion explosive production formula. In this paper, ultrasonic treatment of Fe^{2+} emulsified matrix is used to simulate the demulsification of real emulsified matrix under ultrasonic action, so as to discuss the stability of emulsion explosive.

2. Experiment

2.1. Experimental reagents and instruments

Reagent: water-losing sorbitol monoleate (span-80); Polyisobutylene succinimide (T-152); Compound wax; Nitric acid by; Sodium nitrate; Water; 37% neutral formaldehyde solution; Ferrous sulfate; 0.5mol/L sodium hydroxide standard solution; 1% phenolphthalein indicator.

Instrument: XO-1800D Ultrasonic Cell Crusher: Nanjing Xianou Instrument Manufacturing Co., LTD. JJ-1 precision electric mixer: Jintan Youlian Instrument Research Institute; Electronic balance; 10mL pipette; 50mL basic burette, 1000mL volumetric bottle, 250mL conical bottle, iron stand, 100mL

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measuring cylinder, 250mL beaker, rubber head eyedropper, glass rod, etc.

2.2. *Sample preparation of emulsified matrix*

Under actual laboratory conditions, according to the designed emulsion matrix formula, formula and emulsifier mix as shown in Table 1 and Table2.According to the formula, the nitric acid, sodium nitrate and water are accurately weighed and heated to 110~120℃ to obtain the aqueous phase; Compound wax, span-80 and T-152 emulsifiers were weighed accurately according to the formula and heated to 100~105℃ to obtain the oil phase. Turn on the JJ-1 precision electric stirrer, pour the aqueous phase into the oil phase slowly and then quickly, stir for 2-3 min to get the latex matrix. Numbered, cool for use.

2.3. Demulsification experiment of emulsified matrix

In the production of emulsion explosive, if the emulsification effect is not good, the system will appear the whole or half of inorganic salt (such as ammonium nitrate) particles which are not coated by surfactant. These particles only temporarily exist in the oil phase. When the water is immersed in these very polar particles, they will quickly dissociate to the ionic state in the water. When ultrasonic aqueous solution by mechanical vibration, water phase inorganic salt particles in suspension vibration with continuous oil phase and relative vibration velocity is different, different inorganic salt particles and friction, meet each other, form a larger volume and quality of inorganic salt very fine droplets, and reduces the colloid emulsion matrix of the interfacial film strength and density, is advantageous to the inorganic salt droplet segregation, finally settlement separation.

Ultrasonic wave is used as the force condition to destroy the colloidal interface membrane of emulsion explosive.The stability of explosive can be evaluated quickly and quantitatively by detecting free ammonium nitrate by formaldehyde titration.

Table 1: The components of sample matrix

Component	Ammonium nitrate	Sodium nitrate	Water	Emulsifier	Wax
Mixture ratio	73.5	10.0	10	2.5	4.0

Table 2: The components of sample matrix

Grouping	1#	2#	3#	4#
Span-80：T-152	1:0	2:1	1:2	0:1

2.4. Experimental Procedure

(1) Take 10g sample and evenly spread it on the bottom of a 250mL dry beaker, and add 100mL deionized water.

(2) As shown in Figure 1, set the parameters of XO-1800D ultrasonic cell crushing apparatus, ultrasonic: 1S, clearance: 1S, temperature: 25℃, power: 1260W, time: 25min. The beaker was placed under the amplitude rod, which was 20mm away from the emulsified matrix. Press the start button.

(3) After the ultrasonic, pour the liquid in the beaker into another dry beaker, and stir the liquid thoroughly. Place 20 ml of liquid in a conical flask and add 2 drops of phenolphthalein indicator.

(4) Add 20mL of 37% neutral formaldehyde solution to the conical flask, shake well, and stand for 5-6min.

(5) Titrate the solution in the conical flask with 0.5 mol/L sodium hydroxide standard solution to a reddish color, 30S without fading, that is, the titration end point.

(6) Calculate the precipitated amount of ammonium nitrate by the formula m ammonium nitrate =(0.5×V×0.08004×100)÷25. V is the volume of titrated sodium hydroxide standard solution, mL.

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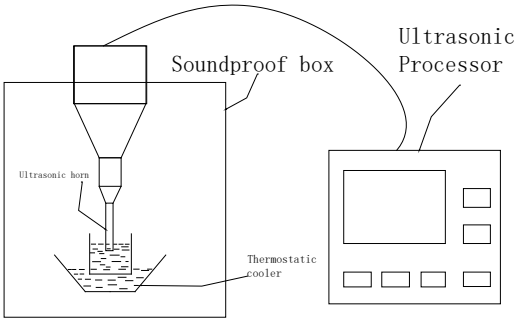

Figure 1: Equipment picture of ultrasonic demulsification.

3. Results and analysis

3.1. Comparison of ammonium nitrate precipitation between ultrasonic forced water solution method and ordinary water solution method

In order to determine whether the ultrasonic force can accelerate the strong polar ammonium nitrate particles to break away from the oil film, 10g of No. 1 ordinary emulsified matrix sample was taken and evenly applied to the bottom of a 250mL dry beaker, and 100mL water was added in the 25℃ water bath for 25min. The test result of ammonium nitrate precipitation was compared with that of the sample under the same condition with ultrasonic force, and the result was shown in Table 3. It can be seen from the test data that the amount of ammonium nitrate precipitated by ultrasonic force is 6~7 times of that of the ordinary water solution method. It can be seen that ultrasonic wave can accelerate the polar ammonium nitrate particles to break away from the oil film and promote demulsification.

Table 3: Comparison between ultrasonic forced method and ordinary water dissolution method.

Project	Dosage of NaOH/ml	Total crystallization amount of 10g matrix/g
Water bath method(25℃、25min)	0.81	0.12966
	0.78	0.12486
	0.79	0.12646
Ultrasonic water bath method (25℃、25min)	5.25	0.84042
	5.24	0.83882
	5.21	0.83402

Figure 2-a shows the emulsified matrix of A1 after ultrasonic treatment for 10min. The surface layer of the emulsified matrix turns from egg yellow to milky white, and the color of egg yellow matrix at the bottom of the emulsified matrix is clearly visible. The emulsified matrix after ultrasonic treatment for 15min (Figure 2-b) has no significant change from that after ultrasonic treatment for 10min. At 20min (Figure 2-c), the thickness of the milky white matrix increased significantly, but a thin layer of egg yellow emulsified matrix was still visible at the bottom of the beaker; at 25min (Figure 2-d), a little egg yellow emulsified matrix was still visible; at 30min (Figure 2-e), the emulsified matrix in the beaker turned completely milky white.

A1-10 *A1-15* *A1-20* *A1-25* *A1-30*

Figure 2: The picture of ultrasonic treatment of A1 emulsion matrix.

As can be seen in Figure 3, when the treatment time increased from 10min to 15min, the precipitation amount of ammonium nitrate increased slightly with time; when the treatment time increased from 15min to 25min, the precipitation amount of ammonium nitrate increased with the increase of the treatment time. As the ultrasonic treatment time continued to increase, the precipitation rate of ammonium nitrate increased.

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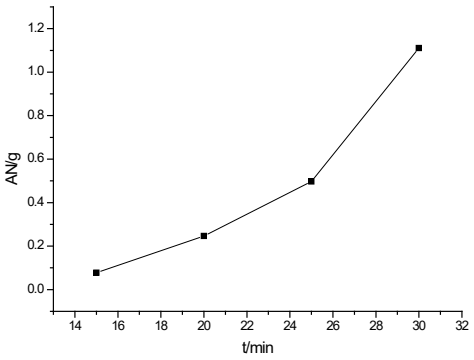

Figure 3: The result of ultrasonic treatment of A1 emulsion matrix.

3.2. Comparative experimental data of demulsification with different emulsified substrates

Apply 10g of emulsified matrix sample evenly to the bottom of a dry beaker of the same specification (200ml) and add 100mL of water. The ultrasonic treatment time was set as 25min. After the set time, the test solution was poured out, numbered, cooled to room temperature, titrated by formaldehyde method. The use amount of sodium hydroxide and the precipitation amount of ammonium nitrate were recorded as shown in Table 4, and the comparison of ammonium nitrate precipitation amount was indicated in Figure 4.

Table 4: The table of titration data.

Groups	Dosage of NaOH/ml		Total crystallization amount of 10g matrix/g	
	Common emulsion matrix(A)	Emulsified matrix containing ferrous iron(B)	Common emulsion matrix(A)	Emulsified matrix containing ferrous iron(B)
1#	5.23	8.64	0.83775	1.38309
2#	2.94	6.79	0.47064	1.08694
3#	0.82	5.22	0.13127	0.83562
4#	—	—	—	—

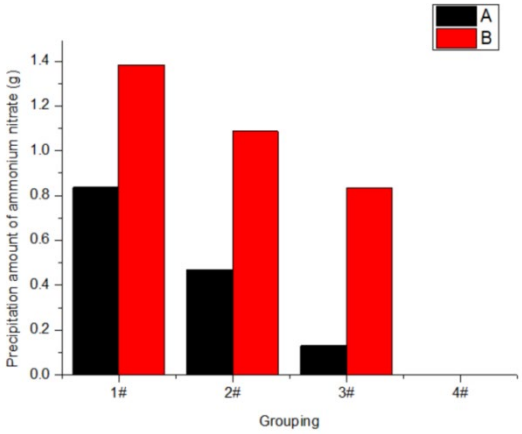

Figure 4: The precipitation amount of ammonium nitrate in different emulsion matrix.

(1) Due to the limitation of experimental conditions, the 4# experiment was not successfully prepared. Compared with Span-80 emulsifier, the oleophilic part of T-152 is located at both ends of the molecule. During emulsification, its polar part extends to the aqueous phase to produce molecular bending, and the two ends extend to the oil phase to produce film, forming stereoscopic obstruction film. Compared between groups A and B, the stability of sample group A is obviously better than that of group B. In group A, 3# precipitated the least ammonium nitrate and the interfacial membrane stability was the best, while in group B, 1# precipitated the most ammonium nitrate and the interfacial membrane stability was the worst. Comparing the emulsified matrix samples in group AB separately, the samples prepared by 1# single emulsifier span-80 were significantly less stable than those prepared by composite emulsifier.

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(2) The emulsified matrix particle is a kind of charged colloid, and the conductivity of the newly emulsified matrix is generally very small, and the conductivity of the emulsified matrix gradually increases with the increase of natural storage time. There is a mutation point during the initial demulsification of emulsified explosive, and the value of the conductivity mutates. After ultrasonic treatment, the oil-water interface film and oil film of emulsified matrix are damaged, and the compactness and strength are reduced, so that the water phase is easy to escape from the coating of oil phase. The more ammonium nitrate precipitated, the worse the stability of the emulsified matrix sample. By comparing the conductivity values of different emulsified substrates (Table 3) with the amount of ammonium nitrate precipitated from the emulsified substrates after ultrasonic treatment (Table 4), the experimental results obtained by ultrasonic method were consistent with the results of interface stability of emulsified substrates reflected by the conductivity data after ten cycles of high and low temperature.

(3) According to the comparison Figure 4, under the same conditions, Fe^{2+} can increase the precipitation rate of ammonium nitrate by about 50%.

4. Conclusions

(1) After 25min interaction between ultrasonic method and 25℃ water bath, it was found that compared with water bath method, ultrasonic method could accelerate the release of ammonium nitrate particles from the oil film, and it was feasible to evaluate the stability of emulsified matrix by ultrasonic method.

(2) Under the same ultrasonic condition, Fe^{2+} can increase the precipitation rate of ammonium nitrate by about 50%.

(3) According to the experimental results, the composite emulsifier is better than Span-80. The explosive matrix produced by composite emulsifier is more stable than that by single emulsifier span-80. The stability of the emulsified matrix is the best when span-80 and T-152 are mixed at a ratio of 1:2.

(4) The best formula of emulsified matrix obtained in this experiment: ammonium nitrate 73.5%, sodium nitrate 10%, water 10%, compound wax 4%, span-80 0.83%, T-1521.67%.

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