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Synthesis and Characterization of Pressure Resistant Agent with Double-Layer Core/Shell Hollow-Structure for Emulsion Explosive

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Abstract: A novel pressure resistant agent (PRA) for emulsion explosive was prepared by a high-speed shear mixer with cenospheres, $Al_2(SO_4)_3$, $NaHCO_3$ and hydrophobic nano-silica as the raw materials. The structural features of the PRAs were characterized using a scanning electron microscope, a laser particle size analyzer and an optical microscope, and the experimental results showed that the PRAs were ellipsoidal microspheres with an average particle size of 200 μm and had double-layer core/shell hollow-structures. The inner hollow shell of the microsphere could serve as sensitizing bubble when it was undamaged whatever the status of outer shell, and once it was broken, the materials in the outer and inner shells would react with each other and generate new sensitizing bubbles in emulsion explosive. The brisance and underwater explosion experimental results showed that when the addition of PRAs was determined to be 2 mass%, the resistance to dynamic pressure of emulsion explosive sensitized by glass microsphere (GM) would be improved significantly without affecting its power. The lead column compression of GM-2 mass% PRAs sensitized emulsion explosive was 16.05 mm, which was close to that of GM sensitized emulsion explosive. When compressed by a RDX booster underwater at the distances of 25 and 75 cm, the dynamic pressure resistance ability of GM-2 mass% PRAs sensitized emulsion explosive would increase by 56.54 and 62.07%, respectively, compared to that of GM sensitized emulsion explosive at the same compression distance. This novel PRA provided double protections for improving the pressure resistant properties of emulsion explosives, especially in the condition of dynamic pressure compression.

Keywords: Emulsion explosive · Pressure resistant · Sensitizer, Microspheres · Hot spot

1 Introduction

Emulsion explosive is mainly composed of emulsion matrix and sensitizer. The sensitizer forms "hot spots" under the action of shock wave and triggers the detonation reaction of emulsion explosive. However, there are two cases that may affect the blasting effect of emulsion explosives. One case is in the deep hole blasting, the shock waves in the air of the hole may be faster than that in the emulsion explosives, and the emulsion explosive at the bottom of the hole would be compressed (channel effect); the other case is in delay blasting, shock waves produced by the earlier detonating emulsion explosive would produce a dynamic load on the unexploded emulsion explosive (dynamic pressure desensitization) [1]. In the blasting operations, we usually find many emulsion explosives undetonated, especially at the bottom of deep hole or in delay blasting operations. These problems have attracted much attention of scholars of industrial explosives all over the world. Many researchers had investigated the detonation characteristics changes of emulsion explosive sensitized by glass microspheres after compression [2–5]. Wietand [6] studied the relative sensitivity of permissible explosives to dynamic pressure passivation by designing an explosive nozzle device that could be destroyed. Nie [7] explored a method to test the detonation performances of the emulsion explosives under dynamic pressure, and then studied the pressure desensitization phenomenon of emulsion explosives as well as numerical simulation on the effect of bubbles sensitization. Sumiya [8] studied the detonation performances of emulsion explosives sensitized by glass microspheres and resin micro-

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spheres under the action of underwater shock wave pressure. However, the above studies were mainly focused on the phenomena of pressure desensitization, test methods and influencing factors, but specific solutions have not been proposed. Cheng [9,10] added the hydrogen storage materials MgH_2 as an energetic sensitizer into the emulsion matrix, and MgH_2 particles could produce H_2 to weaken destruction effect on sensitization bubbles under external pressure, which enhanced the pressure resistant properties of the emulsion explosive. However, sensitization bubbles were easy to become oversized due to the aftereffect of chemical sensitizing by this method, which would affect the detonation performance of the emulsion explosive.

Many studies had proved that the destruction of sensitization bubbles was the main cause of pressure desensitization for emulsion explosives [11,12], therefore, the key issue for improving the pressure resistant ability of emulsion explosive is to reduce the loss of sensitization bubbles. Cenosphere is a kind of hollow fly ash that can float on the water. It can be used as physical sensitizer for emulsion explosives and acted as "hot spots" in emulsion explosives [13]. $NaHCO_3$ is often used as a chemical foaming agent for emulsion explosives, which can form evenly distributed CO_2 bubbles in the emulsion matrix due to its decomposition [14]. "Dry water" is a special encapsulation product, which can be produced by a simple mixing process using hydrophobic nano-silica, and the final product is a spherical carrier system because various active agents can be added into the water phase [15,16]. In the study, a novel double-layer core/shell hollow-structure microsphere was designed and prepared based on the working principle of the foam extinguisher, in which a large amount of CO_2 would be generated rapidly by mixing the two solutions of $Al_2(SO_4)_3$ and $NaHCO_3$ [17]. The $NaHCO_3$ powders and cenospheres that partially loaded with $Al_2(SO_4)_3$ were added into the water phase of "dry water" and mixed by a high-speed shear mixer to form double-layer core/shell hollow-structure microspheres. At a certain range of pressure, the microspheres with double-layer core/shell hollow-structure could separate $Al_2(SO_4)_3$ solid from $NaHCO_3$ solution. The inner hollow shell of the microsphere could serve as sensitizing bubble when it was undamaged whatever the status of outer shell. When the external pressure exceeded the limitation that the microspheres could bear, the inner and outer shell of these microspheres would be crushed and the reaction between $Al_2(SO_4)_3$ and $NaHCO_3$ solutions took place, which produced many tiny CO_2 gas to introduce new sensitization bubbles in emulsion explosive. Hence, this kind of double-layer core/shell hollow-structure microspheres could be used as PRAs for emulsion explosives to improve pressure resistant properties.

2 Experimental Section

2.1 Materials

Cenospheres (average particle size was 93 μm, Henan Yixiang New Material Co., Ltd, China), glass microsphere (average particle size was 55 μm, bulk density was 0.25 g/cm^3. Minnesota Mining and Manufacturing Company, USA), aluminium sulfate ($Al_2(SO_4)_3$, AR), photoinitiator (2-Methyl-1-[4-(methyltho)phenyl]-2-morpholino-1-propanopne, AR), methyl methacrylate (MMA, AR, 99%), hydrophobic nano-silica (SiO_2, AR), sodium bicarbonate ($NaHCO_3$, AR), hydrofluoric acid (HF, AR), were purchased from Shanghai Macklin Biochemical Co., Ltd. China, deionized water (homemade), the emulsion matrix, with a density of 1.31 g/cm^3, was prepared in the laboratory. The composition of the emulsion matrix used in these experiments was described in Table 1.

2.2 Preparation of PRAs

The synthesis of PRAs was mainly divided into five steps (as illustrated in Figure 1). The first step was screening of cenospheres with deionized water to remove the broken and solid particles (see Figure 1a). The second step was perforating of cenospheres with 250 mL of 0.6 mol/L HF etching

Table 1. Composition of emulsion matrix.

Component	NH_4NO_3	$NaNO_3$	$C_{18}H_{38}$	$C_{12}H_{26}$	$C_{24}H_{44}O_6$	H_2O
Mass ratio (%)	75	10	4	1	2	8

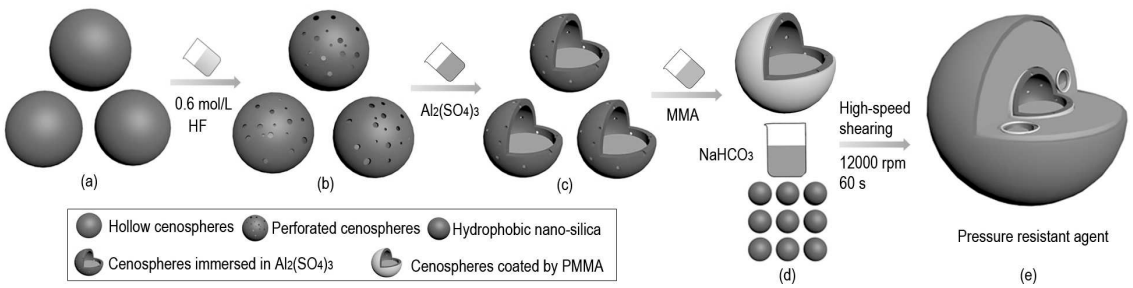

Figure 1. Schematic diagram of the preparation for PRAs.

solution, and the cenospheres with micro/nano-scale holes in the shell were obtained after filtration, washing and drying (as shown in Figure 1b). The third step was loading $Al_2(SO_4)_3$ into the perforated cenospheres via a vacuum impregnation process [18], as elaborated in Figure 2, and then stopped the vacuum operation and opened the separatory funnel valve to force the $Al_2(SO_4)_3$ solution into the hollow space of the cenospheres (as shown in Figure 1c). The fourth step was sealing $Al_2(SO_4)_3$ loaded cenospheres with polymethyl methacrylate (PMMA) [19], and PMMA films were formed on the surfaces of the perforated cenospheres by strong ultraviolet light irradiation of a ultraviolet ray curing machine (1 KW230MM220 V, BLTUV, China), as shown in Figure 1d. The final step was the formation of double-layer core/shell hollow-structure microspheres, where $NaHCO_3$ powder and cenospheres coated with PMMA were added into deionized water to form aqueous phase and then mixed with hydrophobic nano-silica using a high-speed shearing machine (JS30-230, SUPOR, China), and the double-layer core/shell hollow-structure microspheres were obtained after passing a 150-mesh sieve to remove the uncoated cenospheres and the remained hydrophobic nano-silica (see Figure 1e).

2.3 Action Principle of PRAs

PRAs were used as accessory ingredients of sensitizers, and they were distributed uniformly in the emulsion explosive. When the emulsion explosive was suffered from relatively small dynamic pressure, the hollow structure of cenospheres inside the double-layer core/shell microspheres would be undamaged and there were still some gas inside, which could serve as the "hot spots" of emulsion explosive in the detonation process [13]. When the dynamic pressure was beyond the pressure limitation of PRAs, the PRAs and sensitization bubbles in emulsion explosive would be destroyed. Meanwhile, the components of $Al_2(SO_4)_3$ and $NaHCO_3$ inside the PRAs would contact with each other and react quickly to generate CO_2 gas, which could form new sensitization bubbles to improve the pressure resistant properties of emulsion explosive. The chemical reaction formula is as follows:

$$Al_2(SO_4)_3 + 6NaHCO_3 = 3Na_2SO_4 + 2Al(OH)_3 + 6CO_2 \uparrow \quad (1)$$

And in the production of emulsion explosive sensitized by chemical method, the mass ratio of chemical foaming agent $NaNO_2$ of emulsion explosive was normally 0.2 ~ 0.5 mass %, therefore, the mass ratio of $Al_2(SO_4)_3$ in PRA was determined to be 0.6 mass % (the mass ratio of $Al_2(SO_4)_3$ in perforated cenospheres was ca.14 mass %) on basis of gas production.

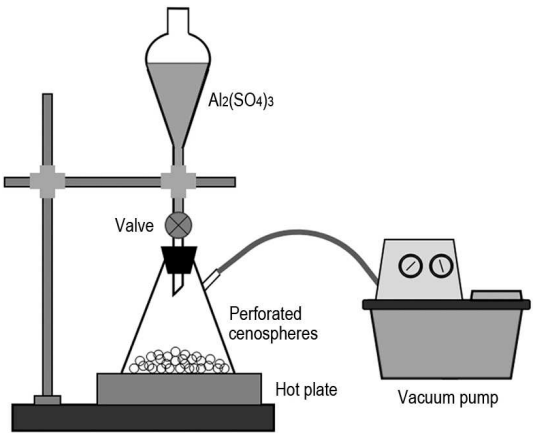

Figure 2. Schematic diagram of vacuum loading device.

2.4 Characterization and Testing

Scanning electron microscope (SEM) and optical microscope (OM) were used to observe the morphology and gas generation process of cenospheres and PRAs. And the particle size distributions of cenospheres and PRAs were measured by a laser particle size analyzer (Mastersizer 2000, Malvern). The explosion power and dynamic pressure resistance ability of emulsion explosive with PRAs were measured by brisance, detonation velocity tests and dynamic pressure desensitization experiments.

3 Results and Discussion

3.1 Microstructure Characterization of PRAs

Figure 3a and b show the untreated spherical cenospheres, whose surfaces were compact and smooth. The cenospheres had a high strength aluminosilicate shell with a thickness of several microns. The shell of cenosphere had a porous structure and covered by an amorphous nano-meter film (see Figure 3c). This special structure guaranteed the amorphous nano-meter film on the surface of cenospheres be etched and dissolved chemically without affecting the mechanical strength of the shell [20, 21]. Figure 3d indicates the cenospheres with many micro-nano level holes on the surface after HF etching solution treatment, so that admixtures could be loaded and released through these pores. Figure 3e shows that the outer surfaces of cenospheres coated with PMMA were obviously pleated compared to the uncoated cenospheres, which indicated that PMMA had uniformly polymerized on the outer surface and encapsulated the cenospheres that loaded with $Al_2(SO_4)_3$ effectively. Figure 3f illustrates that the finally formed PRAs were ellipsoidal with hydrophobic nano-silica shell.

In order to prove that $Al_2(SO_4)_3$ had been successful encapsulated into the perforated cenospheres, the shells of

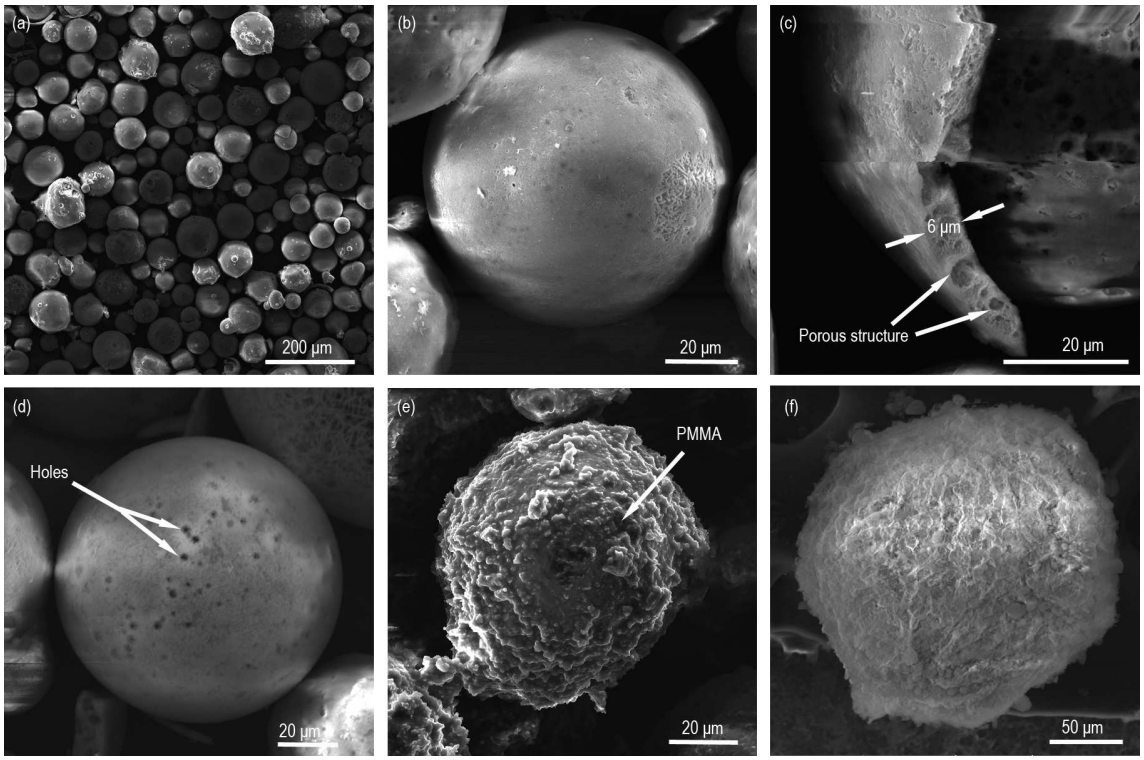

Figure 3. SEM images of: (a) Cenospheres; (b) Cenospheres with impermeable shell; (c) Porous shell of cenospheres, (d) Perforated cenospheres, (e) Cenospheres coated by PMMA, (f) PRAs.

cenospheres before and after loading were pre-treated by squashing operation. Figure 4a shows the smooth inner wall of the cenospheres with a hollow structure (before loading), and Figure 4b demonstrates that the inner wall of the cenospheres of hollow structure was partially deposited with $Al_2(SO_4)_3$ (after loading). The results proved that the method of vacuum impregnation could be used to encapsulate $Al_2(SO_4)_3$ into the cenospheres to form partial hollow microspheres.

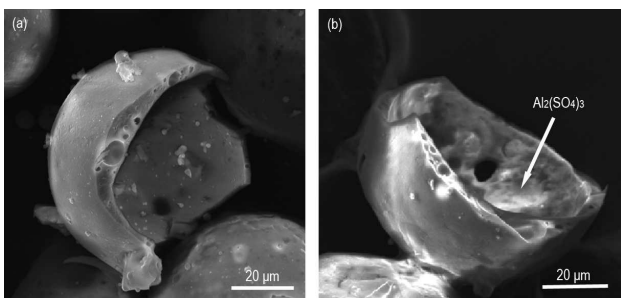

Figure 4. SEM images of: (a) Cenospheres without loading; (b) Cenospheres loaded with $Al_2(SO_4)_3$.

3.2 Particle Size Distribution of PRAs

The particle sizes of the cenospheres (see Figure 5a) and the cenospheres that coated with PMMA (see Figure 5b) were similar, both were ranging from 30 to 250 μm (with an average particle size of 93 μm). The PRA (see Figure 5c) had a much wider particle size distribution and a bigger mean particle size (200 μm).

3.3 Gas Generation Performance Characterization

Figure 6a and b show the OM images of PRAs. In Figure 6b, the brightness difference between inside and outside of the microspheres represents different compositions of PRAs. Furthermore, the gray opaque sphere inside the microsphere was a cenosphere that loaded with $Al_2(SO_4)_3$, which proved that the double-layer core/shell hollow-structure microspheres had been successful prepared. Meanwhile, in order to directly understand the gas generation phenomenon when the PRAs were crushed, a small amount of the PRAs were placed under the optical microscope and crushed with a hard iron spoon. As shown in Figure 6c, many tiny dynamic bubbles were generated from the crushed PRAs, which indicated that the PRAs with double-layer core/shell hollow-structure could generate new sensitization bubbles in emulsion explosives once damaged.

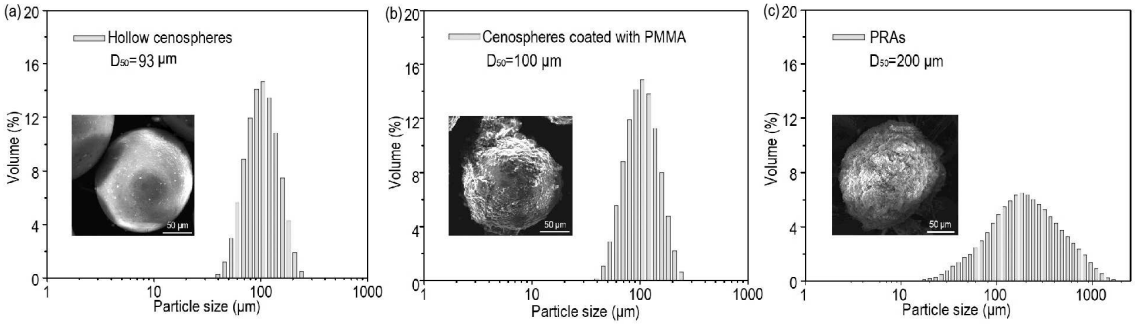

Figure 5. Particle size distributions of: (a) Hollow cenospheres; (b) Cenospheres coated with PMMA; (c) PRAs.

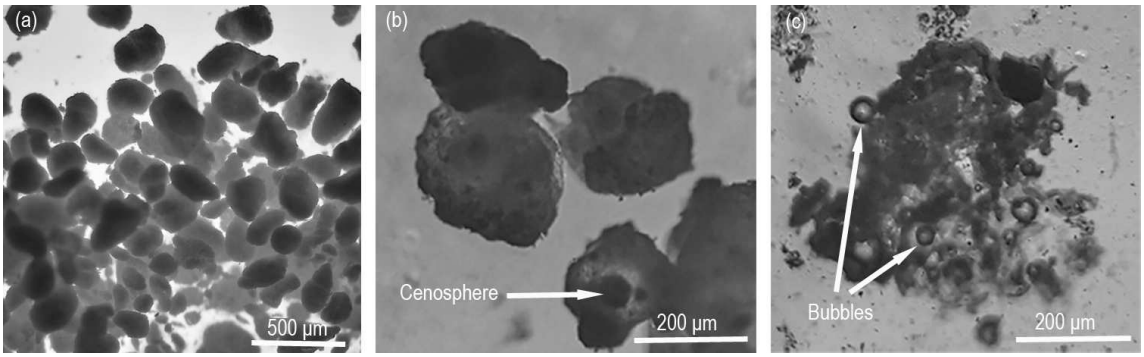

Figure 6. OM images of: (a) and (b) PRAs; (c) Crushed PRAs.

3.4 Explosion Performance Test

The brisance and detonation velocity are very important parameters of explosion power. The influence of different contents of PRAs on the emulsion power of emulsion explosives was studied through the brisance and detonation velocity experiments. In the brisance experiments, the brisance of the emulsion explosive was measured by the Hess test method [22]. The mass of each sample was 50 g and the initial diameter and height of the uncompressed lead column were 40 mm and 60 mm, respectively. In the detonation velocity experiments, the detonation velocity of emulsion explosive sample was obtained using a detonation wave velocity measurer (as shown in Figure 7). The emulsion explosive was filled into a poly vinyl chloride (PVC) tube with the diameter and length of 32 and 270 mm, respectively. The distance between two adjacent metal probes was 50 mm, and the detonation velocity of emulsion explosive sample was recorded using an intelligent detonation velocity instrument (BSW-3 A, China). As shown in Table 2, three kinds of emulsion explosives with different contents of PRAs were prepared and each explosive sample

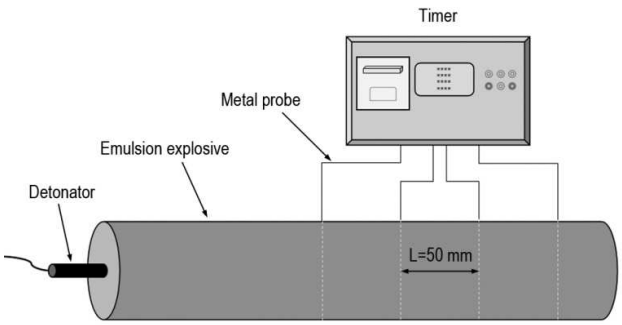

Figure 7. Schematic diagram of detonation velocity test.

Table 2. Formulations and detonation performance of three emulsion explosives.

Emulsion explosives	Mass fraction (mass%) Emulsion matrix	GM	PRAs	Density (g/cm^3)	Brisance (mm)	Detonation velocity (m/s)
GM sensitized	96	4	0	1.18	16.21 ± 0.02	4682 ± 45
GM-2 mass% PRAs	94	4	2	1.15	16.05 ± 0.04	4655 ± 30
GM-4 mass% PRAs	92	4	4	1.11	14.20 ± 0.05	4530 ± 40

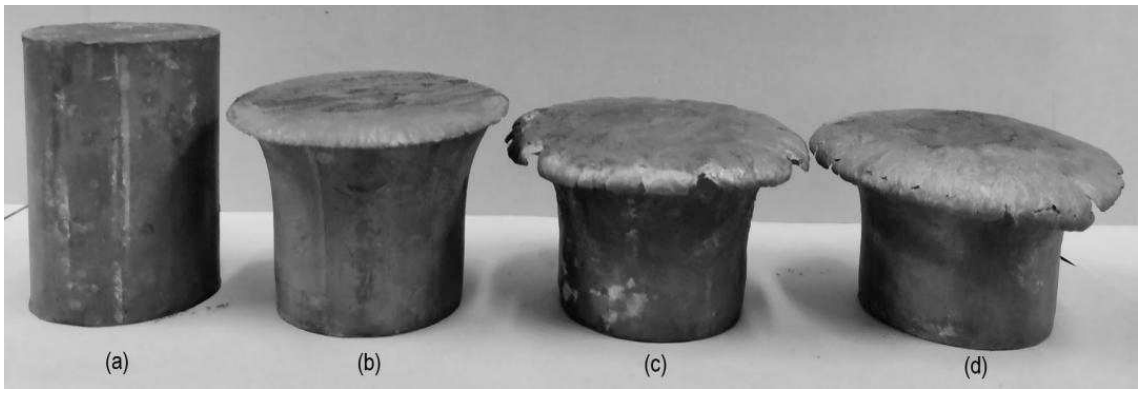

Figure 8. Photos of lead columns: (a) Uncompressed (b) Compressed by GM-4 mass % PRAs sensitized emulsion explosive (c) Compressed by GM-2 mass % PRAs sensitized emulsion explosive (d) Compressed by GM sensitized emulsion explosive.

contained 4 mass % GM. Each kind of sample was tested at least three times and the average value of brisance and detonation velocity was obtained.

Figure 8 and Table 2 show the experimental results of the three kinds of emulsion explosives. The brisance of GM-2 mass % PRAs emulsion explosive was 16.05 mm and only 0.16 mm lower than that of GM sensitized emulsion explosive. While the brisance of the GM-4 mass % PRAs emulsion explosive was 14.20 mm and 2.01 mm lower than that of the GM sensitized emulsion explosive. The detonation velocity of GM-2 mass % PRAs emulsion explosive was 4655 m/s and only 27 m/s lower than that of GM sensitized emulsion explosive. While the detonation velocity of the GM-4 mass % PRAs emulsion explosive was 4530 m/s and 152 m/s lower than that of GM sensitized emulsion explosive.

The explosion power of emulsion explosives decreased with the increasing PRAs. The main reason was that the density of emulsion matrix decreased with the addition of PRAs and the main components of the PRAs were silica and water, which could only act as inert medium in the emulsion matrix and had no effect on improving the explosion power of emulsion explosive [23]. Therefore, the amount of PRAs added in emulsion explosive was determined as 2 mass %.

In order to verify the dynamic pressure resistance ability of emulsion explosive sensitized with 2 mass % PRAs and 4 mass % GM, and the 4 mass % GM sensitized emulsion explosive was compared in dynamic pressure desensitization experiments. Each spherical emulsion explosive sample was around 30 g with a charge diameter of ca. 37 mm. Figure 9a shows the compression system of emulsion explosive. The cylindrical pressed RDX booster was fixed at the center of rectangular steel frame, which was composed of bulk RDX and paraffin with a mass ratio of 100 : 5, and its weight and density were 10 g and 1.65 g/cm^3, respectively. The spherical emulsion explosives were fixed at different distances from RDX with steel wire, and the shock wave generated by the RDX in the center of the device would compress the

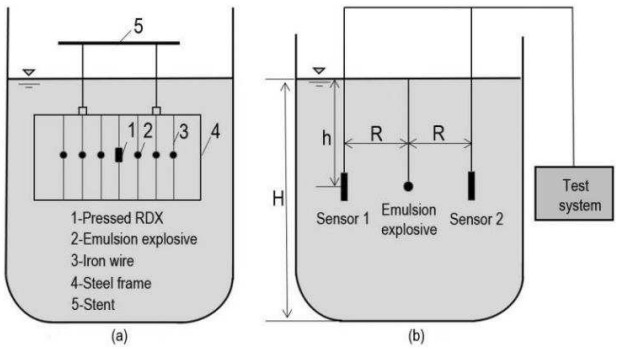

Figure 9. Facilities for testing dynamic pressure resistance of emulsion explosive: (a) Dynamic compression treatment; (b) Underwater explosion experiment.

emulsion explosives with different impact strength, which was used to simulate the dynamic pressure desensitization phenomenon of emulsion explosive in the delayed blasting. Then the explosion performance of these compressed emulsion explosive would be tested by the underwater explosion experiment, as shown in Figure 9b. The distance between the compressed emulsion explosive sample and the sensor (PCB pressure sensor, ICP138 A25, USA) was 0.7 m, and both the emulsion explosive sample and RDX booster were placed 2.5 m below the water surface (the depth and diameter of the underwater explosion testing tower were 5 meters). The compressed emulsion explosive sample was initiated by a detonator and the shock wave signal was recorded using an oscilloscope (Agilent5000 A, USA). The delay time to detonate the compressed emulsion explosives was less than 20 min and each sample was tested three times. In order to quantitatively describe the influence of dynamic pressure desensitization on the detonation performance of emulsion explosive, the parameter "desensitization ratio" was introduced [12]. The calculation formula is as follows:

$$D = (p_0 - p_l)/(p_0 - p_d) \quad (2)$$

Where, D represents the "desensitization ratio"; p_0 and p_l are the shock wave peak pressure of emulsion explosives before and after compression in the underwater explosion experiments, respectively; p_d is the shock wave peak pressure of a detonator in the underwater explosion experiments. Desensitization ratio D equal to zero represents that pressure desensitization has no effect on emulsion explosives, whereas D equal to 100% indicates that the emulsion explosives misfired. When the desensitization ratio D is between 0 and 100%, it indicates that pressure desensitization phenomenon has occurred in emulsion explosives, and the smaller the desensitization ratio, the less the detonation performance of emulsion explosives is influenced by pressure desensitization. In the underwater explosion experiments, the shock wave peak pressures of two detonators were respectively 5.92 and 6.08 MPa, and the average value was 6.0 MPa. By comparing the desensitization ratios of the two kinds of emulsion explosives, the pressure desensitization resistance of the two kinds of emulsion explosives could be obtained. Table 3 shows the underwater shock wave peak pressures and desensitization ratios of the emulsion explosives compressed at different compression distances.

Figure 10 shows the pressure-time curves of the two emulsion explosives obtained from the underwater explosion experiments. The shock wave peak pressures of the two emulsion explosives were reduced after compression, and the peak pressure of GM-PRAs emulsion explosive was higher than that of GM sensitized emulsion explosive at the same compression distance. Table 3 shows that when compressed by shock waves of the same impact strength, the pressure desensitization ratios of GM-PRAs sensitized emulsion explosives were much lower than that of GM sensitized emulsion explosives. When the compression distance was 25 cm, the pressure desensitization ratios of GM sensitized emulsion explosive and GM-PRAs sensitized emulsion explosive were 93.91 and 37.10%, respectively. When the compression distance reached 75 cm, the pressure desensitization ratio of GM sensitized emulsion explosive was still as high as 72.17%, while only 10.10% as for the GM-PRAs sensitized emulsion explosive. It indicated that the addition of PRAs could obviously alleviate the dynamic pressure desensitization problem of emulsion explosive, and the reason was that the destruction of sensitization bubbles was the main cause of pressure desensitization of emulsion explosives [9]. Through previous experiments we found that many GMs in emulsion matrix were crushed after dynamic pressure compression, which led to the reduction of effective "hot spots" in emulsion explosive, so the detonation

Table 3. Shock wave peak pressures and desensitization ratios of emulsion explosives compressed at different distances.

Compression distance (cm)	GM sensitized p_1(MPa)	D (%)	GM-PRAs sensitized p_1(MPa)	D (%)
25	6.7	93.91	12.2	37.37
75	9.2	72.17	14.9	10.10
Uncompressed	17.5	0	15.9	0

Note: p_1 represents shock wave peak pressure, D represents desensitization ratio.

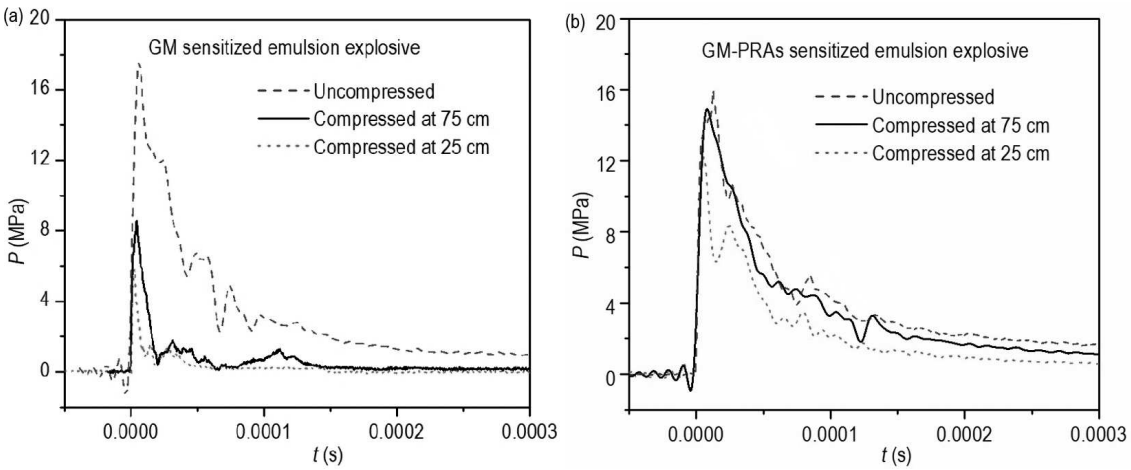

Figure 10. Pressure-time curves of compressed emulsion explosives: (a) GM sensitized emulsion explosive (b) GM-PRAs sensitized emulsion explosive.

performance was weakened [10, 11]. Although high strength dynamic pressure will result in damages of GMs and PRAs in the emulsion matrix, the PRAs could react rapidly to produce gases once destroyed and form new sensitizing bubbles to act as "hot spots", which could weaken the negative effect of dynamic pressure on the detonation performance of emulsion explosive.

4 Conclusions

A novel double-layer core/shell hollow-structure microsphere called "PRAs" was prepared by a high-speed shearing method, which can be used to improve the dynamic pressure resistance ability of emulsion explosives. The inner hollow shell of the PRAs could serve as sensitizing bubble when it was undamaged whatever the status of the outer shell. When the external pressure exceeded the limitation that PRAs could bear, the inner and outer shell of these microspheres were crushed and the reaction between $Al_2(SO_4)_3$ and $NaHCO_3$ solutions took place, then new sensitization CO_2 bubbles would be introduced to improve the pressure resistance ability of emulsion explosive under dynamic pressure.

Brisance and detonation velocity experimental results showed that the explosion power of emulsion explosive decreased with the increasing PRAs, but the brisance and detonation velocity of GM-2 mass% PRAs sensitized emulsion explosive were 16.05 mm and 4655 m/s, respectively, which were very close to GM sensitized emulsion explosive.

Dynamic pressure desensitization experiments showed that when compressed by shock waves of the same impact strength, the pressure desensitization ratio of GM-PRAs sensitized emulsion explosives were much smaller compared to that of GM sensitized emulsion explosives, which indicated that the addition of PRAs could significantly alleviate the dynamic pressure desensitization problem of emulsion explosive.

Acknowledgments

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