United States Patent

Edwards

[15] **3,653,996**

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[54] CONTROLLED GELATION IN AQUEOUS EXPLOSIVES CONTAINING BORIC ACID

[72] Inventor: **Donald W. Edwards,** Lehighton, Pa.

[73] Assignee: **Atlas Chemical Industries, Inc.,** Wilmington, Del.

[22] Filed: **Jan. 22, 1970**

[21] Appl. No.: **5,144**

[52] **U.S. Cl.** **149/21,** 149/2, 149/38, 149/39, 149/40, 149/41, 149/42, 149/43, 149/44, 149/47, 149/57, 149/60, 149/61, 149/62, 149/76, 149/77, 149/78, 149/89, 149/92, 149/105

[51] **Int. Cl.** **C06b 19/04**

[58] **Field of Search** 149/92; 149/2, 21, 38, 41, 149/44, 46, 47, 56, 57, 59, 60, 89, 92, 39, 40, 42, 43, 61, 76, 77, 78, 105

[56] **References Cited**

UNITED STATES PATENTS

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Primary Examiner—Leland A. Sebastian

Attorney—Kenneth E. Mulford, Roger R. Horton and Ernest G. Almy

[57] **ABSTRACT**

Explosive compositions containing boric acid, a gelling agent, an oxidizer, a fuel component, and an aqueous medium and a method of controlling the degree of gelation of these compositions. The compositions can be gelled to the extent desired by adjusting their pH.

27 Claims, No Drawings

CONTROLLED GELATION IN AQUEOUS EXPLOSIVES CONTAINING BORIC ACID

This invention relates to explosive compositions which are capable of controlled gelation and to a method of controlling the degree of their gelation.

Slurry explosives generally contain a gelling agent such as guar gum to prevent the insoluble components of the explosive from segregating, which would make the explosive insensitive. A cross-linking agent is usually also present to cross-link the gelling agent and form a stable gel; generally, borates and chromates are used as cross-linking agents. These compounds form cohesive gels between a pH of about 5 to about 6. Below about 5 only a thickened mass is produced, and above about 6 the gel becomes so tight that the water is squeezed out. With certain types of gums, a gel cannot be formed at all even at a high pH. But even at a pH of 5 to 6, these gels are not pourable and there is no way to control or vary the degree of gelation. As such, they are difficult to load into a borehole and do not adequately fill all the voids in the borehole, which may result in an inefficient utilization of the explosive.

Since most gelling agents require time to gel, the commercial practice is to use excess gelling agent so that the gelation is complete enough at the end of the mixing stage to prevent segregation of the components. However, in addition to producing gels which are not pourable, this practice may also produce gels which are so "tight" that the water is squeezed out leaving the explosive insensitive.

An explosive composition can be gelled with small amounts of gelling agents by heating during the mixing stage in an attempt to enhance cross-linking of the gel prior to storage. Normally, heat alone will not complete all of the cross-linking available from the conventional gelling agents present in the formulation. Thus, additional cross-linking takes place during storage with the possibility of ingredient separation due to the incomplete initial gelation.

ADVANTAGES OF THE INVENTION

I have discovered explosive compositions which can be made to have any desired degree of gelation simply by controlling their final pH. A gelled explosive composition can be made which is tight enough so that the components of the explosive do not segregate on storage, yet is pourable and has good bag release, and the preferred compositions do not gel significantly further on storage. Moreover, gels can be formed at a pH as low as 5 in the compositions of this invention using as gelling agents those gums which would previously gel only at at higher pH.

But the most remarkable property of the compositions of this invention is the ease with which the degree of gelation can be controlled. While prior art gels, once formed, cannot be easily "ungelled," the gels of this invention can be repeatedly made tighter or weaker by successive adjustments of their pH.

COMPOSITIONS OF THIS INVENTION

The explosive compositions of this invention comprise a gelling agent, boric acid, an aqueous medium, an oxidizer, and a fuel component. An explosive composition may be formed from 1 part by weight (pbw) oxidizer, about 0.14 to 0.43 pbw water, about 0.007 to 0.4 parts per part of water of a two-component mixture about 20 to 80 percent of which is a boric acid and about 20 to 80 percent of which is a gelling agent and a sufficient amount of a fuel component to give an oxygen balance between about +10 and −30. The explosive composition also preferably contains about 0.7 to 2.5 pbw of a sensitizer.

Boric acid is the cross-linking agent and is the component of the compositions of this invention which gives them their unusual properties. As smaller and smaller amounts of boric acid are used, the gel becomes thinner and thinner; the full range of gelation from a very thin to a tight gel may be obtained if the amount of boric acid used is equal to at least about 25 percent of the amount of gelling agent used. Excessive amounts of boric acid do not appear to be harmful, but merely unnecessary.

The boric acid is preferably obtained directly from boric acid, $B(OH)_3$, but may also be obtained from boron compounds which can be substantially dissociated into boric acid. Such compounds include borax, metaboric acid and the metaborates, alkyl boric acids such as butylboric acid, aromatic boric acids such as phenyl boric acid, sodium tetramethoxyborate (borine), boron halides such as boron trichloride and boron tribromide, and boron sulphide.

The gelling agent is a substance, usually polymeric, which is capable of being cross-linked in water to form a gel which is stable in the presence of the oxidizer. The gelling agent is preferably a polysaccharide, preferably a galactomannan such as locust bean gum or guar gum, but may also be a gelling agent such as a biopolymer. Guar gum is usually preferred because of its availability.

The oxidizer is the source of oxygen during the explosion and is preferably ammonium nitrate. However, other inorganic oxidizer salts such as sodium nitrate, calcium nitrate, magnesium nitrate, potassium nitrate, lithium nitrate, ammonium chlorate, potassium chlorate, sodium chlorate, ammonium perchlorate, potassium perchlorate, or sodium perchlorate may also be used.

The fuel component is a substance which consumes oxygen during the explosion. This definition would include the components which function as a gelling agent as well as many components which function as sensitizers. In addition, materials may be added primarily for the purpose of consuming oxygen such as sulfur, coal, bagasse, fuel oil, paraffin oil, etc.; sulfur is preferred. Liquid fuels such as ethylene glycol, formamide, or glycerine may also be used either to reduce the amount of water or to promote low-temperature pourability.

A sensitizer is a substance which increases the sensitivity of a composition and includes explosive compounds, air-entrapping materials, and finely divided metals. Explosive sensitizers include, for example, nitroglycerin, TNT, DNT, tetryl, RDX, PETN, nitromethane, and amine nitrates such as methyl amine nitrate and ethylene diamine dinitrate. Examples of air-entrapping materials include expanded perlite and glass, urea-formaldehyde and phenol formaldehyde micro-spheres. Aluminum and magnesium, for example, may be used as finely divided metal sensitizers. A combination of an air-entrapping material and an explosive sensitizer is preferred because it produces the greatest sensitivity.

Other compounds may also be included in the compositions for various purposes, such as surfactants to help promote the gelling process.

METHOD OF CONTROL

The present invention contemplates control over the degree of gelation of gelling agent, water, and boric acid compositions by adjusting the pH of the composition. Essentially, one first prepares a mixture containing at least the boric acid, the gelling agent, and the aqueous medium, although the mixture may also contain other components of the composition. The pH is adjusted to the final pH with a base or an acid, for example, ammonium hydroxide or acetic acid, and any remaining components are mixed in. Alternatively, the boric acid may be added after the pH is adjusted to the final pH, but it is preferably to add the boric acid before the final pH adjustment so that one may observe the degree of gelation as one adjusts the pH.

The final pH determines the properties of a given composition. The higher the final pH, the tighter will be the gel, and the lower the final pH, the more fluid will be the gel; also, the gel is more adhesive at lower pH levels and, therefore, will give better release from a plastic bag at higher pH levels. It is preferable to start at a low pH, say, at most about 4.5, and to raise it to the final pH rather than starting at a high pH and lowering it to the final pH because it is much easier to disperse any insoluble materials in a thin gel than in a tight gel.

Generally, a final pH above about 7 is undesirable because the gel is so tight that water squeezes out, the components separate, and it is not pourable and has no water-resistance; however, it is still detonable. Also, a final pH less than about 2 is generally undesirable because a thickened mass results rather than a gel; it has no water-resistance either but is still detonable. For best results, the final pH should be between 4.5 and 7, although these limits will vary with the composition used; a pH between 5.0 and 6 is the preferred range.

If the composition contains a liquid which is not easily gelled such as some oily fuels, for example, fuel oil, paraffin oils, and particularly nitroparaffins such as nitromethane, the best procedure is to prepare a mixture containing at least the boric acid, the gelling agent, the aqueous medium, and the liquid not easily gelled. The pH is lowered to about 1.5 to 4.4, and the mixture is blended well and preferably heated. The pH is then adjusted to the final pH.

Generally, heating during mixing is desirable as it decreases the time necessary to form a gel; 100° to 180° F. is a suitable range but 140° to 150° F. is preferred If explosive components are used which are heat-sensitive, the composition should either be heated and cooled before the explosive components are added or should not be heated.

It is also preferable to add any large, solid materials such as prills after the final pH adjustment since the gel has formed by then and will prevent their segregation.

If a boron compound which dissociates into boric acid under the conditions of this invention is used, the boron compound should be added to the gelling agent under conditions which prevent its reaction with the gelling agent prior to dissociation. Some boron compounds such as the metaborates dissociate rapidly enough that the conditions of reaction are sufficient for their use and no special conditions are necessary. Others such as borax and the biborates require time to dissociate and should be acidified prior to being mixed with the gelling agent. In addition, a composition using borax may tend to vary in pH with time and require adjustments to keep the pH below 7.

EXAMPLES

Tables I and II below show the types of gelation which resulted when various compositions were prepared and stored:

COMMENTS FOR TABLES I AND II

- A Would not form a gel
- B Thickened mass
- C Thin, non-cohesive gel
- D Thin, pourable gel
- E Loose, cohesive gel; one hour, very tight gel
- F Pourable cohesive gel system
- G Semirigid gel
- H Tight gel – not pourable

1. Provided by Stein, Hall & Co., Inc. under the trademark "Jaguar DE-1"
2. Provided by Stein, Hall & Co., Inc. under the trademark "X0589"
3. Sold by General Mills, Inc. under the trademark "X0589"
4. Sold by Stein, Hall & Co., Inc. under the trademark "705D-AA"
5. An anionic heteropolysaccharide produced by the fermentation of a carbohydrate by the bacterium Xanthomonas campestris and sold by General Mills, Inc. under the trademark "XB-23"
6. Sold by Stein, Hall & Co., Inc under the trademark "Polyhall M-295"
7. Sold by Oil Center Research, Inc. under the trademark "TX-150" and determined by analysis to be about 33.5 percent boric acid, about 35.8 percent guar gum, about 22.9 percent water, about 6.7 percent polyacrylamide, and about 1.1 percent ester, probably triglyceride

The compositions A to R of Tables I and II were prepared by mixing the ammonium nitrate and ethylene diamine dinitrate solutions, heating to 140° to 150° F. (except composition H where heating was not used), and mixing into the solution a pre-mix of all the other ingredients. The pH of the resulting mixture was adjusted with acetic acid to about 3.5 and then raised with ammonium hydroxide over a 2-minute period with stirring until the gel formed. Composition S was prepared in the same manner except that the boric acid was added after the final pH adjustment. The compositions were then stored at room temperature for various periods, and the tests were terminated at the last reading. The ammonium and sodium salts of boric acid were prepared by neutralizing a saturated solution of boric acid with ammonium and sodium hydroxides to a pH of 10 to 11, respectively, and evaporating to dryness; these salts apparently did not immediately revert to boric acid in solution (compositions O and P).

TABLE I

Composition, percent	A	B	C	D	E	F	G	H	I	J
60% Ammonium nitrate solution	77.05	77.05	77.05	77.05	77.05	77.05	77.05	77.05	77.05	77.05
50% Ethylene diamine dinitrate solution	21.25	21.25	21.25	21.25	21.25	21.25	21.25	21.25	21.25	21.25
Boric acid							0.78	0.71	0.69	0.69
Ammonium biborate ($NH_4HB_4O_7.3H_2O$)				0.69						
Borax ($Na_2B_4O_7.10H_2O$)					0.69					
Acidified borax						0.78				
Non-ionic Guar gum [1]	1.42			0.85	0.85	0.92	0.92	0.83	0.16	
Modified Guar gum		[2] 1.70								[3] 0.16
Polysaccharide [4]			1.7							
Bio-polymer [5]									0.85	0.85
Polyacrylamide [6]	0.28			0.16	0.16			0.16		
Final pH	5.7	6.1	5.75	6.2	6.0	6.15	5.5	5.5	5.9	5.9
Fresh	H	D	C	A	E	F	F	F	F	F
7 days		C				F			H	G
10 days			C							
50 days			C							
90 days	B						F			
120 days								F		

TABLE II

Composition, percent	K	L	M	N	O	P	Q	R	S
60% Ammonium nitrate solution	77.05	77.05	77.05	77.05	77.05	77.05	77.05	77.05	77.05
50% Ethylene diamine dinitrate solution	21.25	21.25	21.25	21.25	21.25	21.25	21.25	21.25	21.25
Boric acid	0.78	0.78	0.78					0.78	0.78
Ammonium salt of boric acid					0.78				
Sodium salt of boric acid						0.78			
Sodium metaborate ($Na_2B_2O_4.8H_2O$)							0.78		
Boric Acid-Gum-Amide-Ester Mixture [7]				1.7					
Non-ionic Guar gum					0.92	0.92	0.92	0.92	0.92
Modified Guar gum	[7] 0.92								
Locust Bean gum		0.92							
Polysaccharide [4]			0.92						
Polyacrylamide [6]									
Final pH	6.05	6.1	5.75	5.75	6.3	6.3	5.9	5.75	5.75
Fresh	F	F	F	F	H	H	F	F	F
7 days	F	F			F	F	F		
10 days			F						
50 days			D	F				C	F

As to the compositions of this invention (F to S), the rise in pH produced a corresponding increase in the degree of gelation. As to composition E, once a gel had formed, a further increase in pH had no noticeable effect. Table I shows, inter alia, that the preferred composition H withstood 120 days of storage without further gelation occurring, while prior art composition E formed a tight gel within an hour. Composition E contrasts markedly with composition F where the borax was dissolved in water and acidified with nitric acid to a pH of about 1.0 to dissociate it into boric acid before it was added to the gelling agent. Although the non-preferred compositions of this invention (I and J) did not exhibit extended storage stability, their degree of gelation was nevertheless pH sensitive.

Another composition identical to composition R was prepared in the same manner as composition S. The pH of this composition was then successively adjusted up and down with acetic acid and ammonium hydroxide. The following degrees of gelation were observed at successive pH values:

pH	Degree of Gelation
3.9	Thickened mass
4.7	Loose gel, very tacky
5.3	Excellent pourability, tacky (poor release)
5.8	Excellent pourability, good release
6.0	Good pourability, excellent release
6.4	Slightly tight gel, poor pourability, excellent release
6.6	Tight gel, not pourable, breaks apart easily
6.0	Good pourability, excellent release
5.25	Excellent pourability, tacky
4.9	Loose gel, very tacky
4.5	Thickened mass
6.9	Tight gel, not pourable, breaks apart easily, water squeezed out
6.0	Good pourability, excellent release
4.75	Loose gel, very tacky
5.7	Excellent pourability, good release
3.5	Thickened mass
5.75	Excellent pourability, good release

Table III below shows additional explosive compositions prepared according to this invention.

Composition L was prepared by mixing the nitrates, dinitrate, and the water, heating to 160° F., and adjusting the pH to 4.0 with acetic acid. The air-entrapping spheres, sulfur, gelling mixture, and boric acid were blended and the heated nitrate solution was added incrementally while the temperature was maintained at 145° F. The surfactant was added and the pH adjusted to 4.5 to 5.0 with sodium hydroxide solution. The nitromethane and ethylene glycol were mixed in and the pH was adjusted to the final pH with sodium hydroxide.

Composition M was prepared by mixing the sodium and ammonium nitrates and water at 150° F. The pH was adjusted to 3 to 4 with acetic acid and the "Jaguar 100" and boric acid were mixed in until thick. The air-entrapping spheres, sulfur, and ethylene diamine dinitrate were mixed in and the pH adjusted to 4 with sodium hydroxide. The ethylene glycol and nitromethane were added and the final pH adjustment was made with sodium hydroxide.

All of the compositions were pourable, and most of the compositions (H, I, and K, for example) are pourable at low temperatures (20° to 30° F.); all the compositions had an oxygen balance between +10 and −30. The compositions were placed in 3 inch diameter plastic cartridges and were detonated with a 3 inch × 6 inch 75 percent gelatin dynamite.

What is claimed is:

1. An explosive composition comprising boric acid, a gelling agent, an aqueous medium, an oxidizer, and a fuel component.

2. A composition according to claim **1** which contains a sensitizer.

3. The composition of claim **1** wherein said boric acid is obtained by acidifying borax.

4. The composition of claim **1** wherein said gelling agent is a polysaccharide.

5. The composition of claim **4** wherein said polysaccharide is a galactomannan.

6. The composition of claim **5** wherein said galactomannan is guar gum.

7. An explosive composition comprising 1 pbw oxidizer, about 0.14 to about 0.43 pbw water, about 0.007 to about 0.4 parts per part of water of a two-component mixture about 20 to 80 percent of which is boric acid and about 20 to 80 percent of which is a gelling agent, and a sufficient amount of a fuel component to give an oxygen balance between about +10 and −30.

8. The composition of claim **7** wherein said boric acid is obtained by acidifying borax.

9. A composition according to claim **7** wherein about 0.007 to about 2.5 pbw of a sensitizer is present.

10. A composition according to claim **9** wherein said sensitizer is a combination of an air-entrapping material and an explosive.

11. The composition of claim **10** wherein said air-entrapping material is phenol formaldehyde or glass microspheres.

12. The composition of claim **7** wherein said gelling agent comprises a polysaccharide.

Composition, percent	A	B	C	D	E	F	G	H	I	I	K	L	M
Ammonium nitrate	41.5	36.2	31.2	40.7	39.2	34.2	32.3	52.9	35.8	39.2	47.05	43.3	42.9
Sodium nitrate	18.0	11.0	11.0	16.0	11.0	11.0	18.0		11.0	11.0		10.0	10.0
Calcium nitrate		5.0	5.0		5.0	5.0				5.0			
Gelling mixture [2]	1.1	0.9	0.9	0.9	0.9	0.9	1.3	1.0	0.75	0.9	0.85	0.6	1.1
Boric acid								0.5	1.0		0.5	0.6	
Water	14.0	20.0	20.0	18.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0
Ethylene glycol		7.5	7.5		7.5	7.5		10.0	10.0	7.5	10.0	5.0	5.0
Surfactant [3]	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.6	0.45	0.4	0.6	0.5	1.0
Sulfur		3.0	3.0	2.0	3.0	3.0	3.0	2.0	3.0	3.0	3.0	3.0	3.0
Aluminum							3.0						
Air-entrapping spheres [1]				2.0	3.0	3.0	2.0	3.0	3.0	3.0	3.0	2.0	2.0
Ethylene diamine dinitrte			5.0		10.0	15.0	20.0	5.0	5.0			5.0	5.0
Nitromethane								5.0	10.0	10.0	15.0	10.0	10.0
Nitrostarch		16.0	16.0	20.0									
T.N.T	25.0												
Final pH	nt	5.55	5.8	5.0	5.95	5.75	nt	5.55	5.7	6.1	5.75	5.85	
Density, g./cc	1.47	1.46	1.44	1.40	1.26	1.28	nt	1.16	1.22	1.25	1.20	1.29	
Velocity, f.p.s	16,200	12,195	11,900	17,090	16,340	16,025	17,045	14,285	17,095	17,360	17,360	17,010	16,670

nt=Not tested.

[1] Phenol formaldehyde in D, E, F, G, I, and J; glass in H, K, L, and M.

[2] 41.7% boric acid, 49.2% guar gum, 9.2% polyacrylamide in A to K; 50–50 mixture of "Jaguar 100" and "Jaguar DE," non-ionic guar gums sold by Stein, Hall & Co., Inc. in L; 50% boric acid and 50% "Jaguar 100" in M.

[3] Polyoxyethylene alkyl amine sold by Atlas Chemical Industries, Inc. under the trademark "G-3780A".

In table III, compositions A to K were prepared by mixing the water, surfactant, and the nitrates and dinitrate at 140° to 150° F. The pH was adjusted to 2 to 3 and the nitrostarch and TNT were added where used; after stirring the aluminum was added (composition G). A pre-mix containing the resin spheres, sulfur, and the gelling mixture was prepared and added with stirring. When the compositions began to thicken, the pH was adjusted with acetic acid to 3.5 to 4.5, and the ethylene glycol and nitromethane were added and stirred in for 1 minute where used. The pH was then adjusted with ammonium hydroxide to the final pH which instantly resulted in the formation of the gel. Whole ammonium nitrate prills were then stirred in, 15 percent (this percentage is included in the figure given in Table III for ammonium nitrate) in cases B and C; 10 percent in case E; and 5 percent in cases H, J, and K.

13. The composition of claim 12 wherein said polysaccharide is a galactomannan.

14. The composition of claim 12 wherein said galactomannan is guar gum.

15. A method of controlling the degree of gelation of an explosive composition comprised of boric acid, a gelling agent, and water, comprising raising the pH of said composition to increase the degree of gelation and lowering the pH to decrease the degree of gelation.

16. The method of claim 15 wherein said pH is adjusted to about 4.5 to 7.

17. The method of claim 16 wherein said composition contains an oxidizer and a fuel component and is detonable.

18. The method of claim 17 wherein said gelling agent comprises a polysaccharide.

19. The method of claim 18 wherein said polysaccharide is a glactomannan.

20. The method of claim 19 wherein said galactomannan is guar gum.

21. The method of claim 17 wherein the pH is adjusted by lowering it to below about 4.5 and then raising it until a gel of the desired consistency is obtained.

22. The method of claim 17 wherein said composition is heated while being gelled.

23. The method of claim 22 wherein said composition is heated to 100° to 180° F.

24. A method of controlling the degree of gelation of an explosive composition which comprises boric acid, a gelling agent, an aqueous medium, an oxidizer, a fuel component, and a liquid not easily gelled comprising preparing a mixture which comprises boric acid, a gelling agent, an aqueous medium, an oxidizer, a fuel component, and said liquid, adjusting the pH of said mixture to between 1.5 and 4.4, blending said mixture well, and adjusting the pH of said mixture to between 4.5 and 7.

25. The method of claim 24 wherein said liquid not easily gelled is a nitroparaffin.

26. The method of claim 25 wherein said nitroparaffin is nitromethane.

27. A method of controlling the degree of gelation of the composition of claim 6 comprising adjusting the pH of said composition.

* * * * *

PO-1050
(5/69)

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,653,996 Dated April 4, 1972

Inventor(s) Donald W. Edwards

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 64, "preferably" should read -- preferable --

Column 4, under section COMMENTS FOR TABLES I AND II, Item 2, "trademark "X0589""should read -- trademark Jaguar EX-JB --

Column 4, under section COMMENTS FOR TABLES I AND II, Item 4, " "705D-AA" " should read -- "705D-A" --

Column 4, line 41, "10 to 11" should read -- 10 and 11 --

The table bridging Columns 5 and 6, at approximately line 41 should be labeled -- TABLE III --.

In said table above (TABLE III) bridging Columns 5 and 6, at approximately line 42 the column headings "H I I K" should read -- H I J K--and so on.

In said table above (TABLE III) bridging Columns 5 and 6, under table heading Composition, percent, "Ethylene diamine dinitrte" should read -- Ethylene diamine dinitrate --.

Signed and sealed this 5th day of December 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

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Commissioner of Patents

PO-1050
(5/69)

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