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"METHACRYLATE INTERPOLYMERS AS GASOLINE THICKENING AGENTS"

by
E. C. Kirkpatrick
E. I. duPont de Nemours and Company, Ammonia Department

Report OSRD No. 3763
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"METHACRYLATE INTERPOLYMERS AS GASOLINE THICKENING AGENTS"

Service Directive CWS-21

Endorsement (1) From E. P. Stevenson, Chief, Division 11 to Dr. Irvin Stewart, Executive Secretary of the National Defense Research Committee.

Forwarding report and noting:

> "Interpolymer gels containing both isobutyl methacrylate and free methacrylic acid, but no sodium soaps, have certain advantages over the isobutyl methacrylate-sodium soap formulas currently used for filling incendiary bombs, but hitherto they have not been sufficiently stable at low temperatures. This report describes interpolymer gels which are stable at low temperatures making them attractive for consideration for filling incendiary bombs."

Forwarding report and concurring.

This is a progress report under Contract 11-364, OEMsr-744 with E. I. duPont de Nemours and Company.

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Abstract

The development of stable isobutyl methacrylate-methacrylic acid interpolymer thickened gasoline gels is reviewed. Laboratory formulation studies on the effect of the nature and the concentration of the polymer, the gelation agent, the soap forming acids, and the fuel composition are described. The evaluation of representative gels as the fillings for large incendiaries are discussed. A soap free gel containing 3 to 6% of an interpolymer prepared from isobutyl methacrylate with 0.3% methacrylic acid, 20% toluene, 1% of 40% aqueous NaOH, and 76 to 73% gasoline gave best results in firing tests in the M-47 and E-9 incendiary bombs. Such a gel has high strength, a rapid burning rate and a high fuel content.

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AMMONIA DEPARTMENT
E. I. DU PONT DE NEMOURS & CO.
****

CHEMICAL DIVISION
DU PONT EXPERIMENTAL STATION
WILMINGTON, DELAWARE
****

April 26, 1944

Isobutyl Methacrylate-Methacrylic Acid Interpolymers as Gasoline Thickening Agents

I. Introduction

Early in 1942 this group cooperated with the Technical Division of the Chemical Warfare Service in a study of incendiary fillings for the M-47 bomb. The gels which gave the best dispersion patterns and which had the best incendiary characteristics were solutions of an isobutyl methacrylate-methacrylic acid interpolymer in gasoline gelled by the addition of aqueous caustic. These gels were not stable at -40°F., however, and they were therefore rejected in favor of more stable mixtures containing both polymer and soaps as represented by the IM-1 and IM-4 formulas (compositions are given in Table 1, page 4).

During the course of a subsequent study of incendiary gels undertaken in this laboratory under OSRD contract OEMsr-744, the stabilization of interpolymer gels was achieved. This report will describe formulation studies on isobutyl methacrylate-methacrylic acid interpolymers in mixtures containing 1% or less soap. Interpolymer gels containing higher concentrations of soap have distinctly different properties and will be reported separately. The interpolymer gels were evaluated in the AN-M47 bomb by the Chemical Warfare Service and in the 40-lb. E-9 incendiary bomb by the Texas Company.

II. Summary

1. Isobutyl methacrylate-methacrylic acid interpolymers were tested as thickening agents for incendiary fillings for large munitions. Laboratory formulation studies on the effect of the nature and concentration of the polymer, the gelation agent, the soap forming acids and the fuel composition, led to the following observations.

a. The strength and elasticity of the gels is proportional to the polymer content and to the methacrylic acid content of the interpolymer. The optimum range appears to be 3 to 6% of an interpolymer of isobutyl methacrylate with from 0.1 to 0.3% methacrylic acid.

b. While all strong bases will produce gelation of the interpolymer solutions, 0.5-1.0% of 40% sodium hydroxide is the preferred gelation agent. Use of a more dilute caustic solution will cause gel friability.

c. The presence of 1% of naphthenic acid reduces the resilience and improves adhesion of interpolymer gels without reducing gel strength. The presence of other soaps stiffens and strengthens interpolymer gels but simultaneously reduces their burning rate and tendency to flow during combustion. The presence of oil-in-water emulsifying agents in interpolymer gels improves stability at -40°F. but produces syneresis at 125°F.

d. The nature of the gasoline markedly affects the strength, stability, and rate of gelation of interpolymer gels. Satisfactory gels were obtained with Purol naphtha meeting CWS Specification 196-131-124, but further work on gasoline specifications is required.

The replacement of 20% of the fuel (gasoline) by toluene results in interpolymer gels stable in one-month surveillance tests at 125° and -40°. The addition of toluene reduces the temperature dependence of viscosity and imparts fluidity without a sacrificing in gel strength. Cosolvents other than toluene either did not improve low temperature stability or caused phase separation at 125°F.

2. The physical properties of isobutyl methacrylate-methacrylic acid interpolymer gels are compared with other accepted incendiary gels. The low soap content of the interpolymer gels results in a rapid burning rate, a high fuel content and a marked reduction in the dependence of consistency on temperature.

3. Ten gels of the type described in this report were submitted for evaluation in firing tests. In the M-47 bomb a gel containing 5% of a 0.1% methacrylic acid - 99.9% isobutyl methacrylate interpolymer had firing characteristics superior to the IM-1 or F-107 gels (Table 1, page 4). The Texas Company's preliminary study of fillings for the 40-lb. E-9 bomb indicates that interpolymer gels will set extremely rapid destructive fires. The preferred compositions contained 4% of a 0.3% methacrylic acid - 99.7% isobutyl methacrylate interpolymer, 20% toluene and, in one case, 1% of naphthenic acid.

III. Recommended Gel Formulas and their Properties

The isobutyl methacrylate-methacrylic acid interpolymer gels listed in Table 1, page 4 are recommended for use as the incendiary filling in large munitions. The optimum interpolymer content will be dependent on the bursting pressure of the particular munition under consideration, but will probably fall in the range of 3 to 6%. Since an increase in polymer content affects the incendiary characteristics of a gel adversely, it is recommended that the polymer concentration be reduced to the minimum compatible with satisfactory dispersion of the incendiary filling. In all cases the optimum toluene concentration is 20% and in the absence of soaps the optimum amount of gelling agent is 1% of 40% aqueous sodium hydroxide.

The advantages of such gels are demonstrated by a comparison of their properties with accepted incendiary gels, as shown in Table 1. The gels containing interpolymer have impact strengths greater than have been observed on any soap fortified AE polymer gels. The low solids content of interpolymer gels results in an extremely rapid burning rate and a high total heat content. The calculation of the BTU content per gallon of gel shown in Table 1 is based on the assumption that straight gasoline contains 126,460 BTU per gallon. The interpolymer gels therefore contain 97 to 99% of this theoretical limit.

	Recommended Interpolymer Gels			Accepted Formulations			
Code	**A3489**	**A3487**	**A3942**	**IM-1**	**IM-4**	**IM-3**	**NP-1**
Composition (wt. %)							
Napalm							13.5
AE Polymer				5	3	2	
0.3% Interpolymer*	4	4	6				
Stearic acid				3	4	3	
Naphthenic acid		1				3	
40% NaOH	1	1.5	1			4.5	
CaO(-40 mesh)				2	4		
H_2O				1.25	2.5		
Gasoline	75	73.5	73	88.75	86.5	87.5	86.5
Toluene	20	20	20				
Physical Properties							
Impact Strength "C"	15.2**	15.2**	17.2**	13.0	14.8	3.6	7.6
Parallel Plate(cm)							
at 25°C.	7.5	10.0	7.4	8.2	7.2	10.4	6.5
at 7°C.	7.1	8.4	7.1	7.4	5.8	9.8	6.3
Stability % Syneresis							
1 mo. at -40°F.	0	0.8	0	2.6	10.2	0.6	0
1 mo. at 125°F.	0	0.4	0	1.0	0.5	2.0	0
Incendiary Characteristics							
Comparative Burning Time minutes	2.7	2.8	3.9	6.4	6.5	5.5	5.8
BTU of fuel/lb. gel	18,700	18,400	18,300	17,800	17,300	17,500	17,300
BTU of fuel /gal.of gel	125,000	123,000	122,000	119,000	116,000	117,000	116,000

*An interpolymer of isobutyl methacrylate with 0.3% methacrylic acid.

**Minimum not true value; see discussion in Appendix.


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IV. Formulation Studies

The composition and physical properties of all interpolymer gels containing no more than 1% soap forming acids, prepared in this laboratory, are shown in Summary Sheet, Table 5 in the Appendix. The only gels which were stable in surveillance were those containing 20% toluene as cosolvent. The properties of the unstable gels, however, are also pertinent, since they bear on the formulation of stable toluene-containing gels. Formulation studies are discussed below in relation to the variable under investigation.

A. Polymer Content and Composition

The strength and elasticity of the gels vary directly with the polymer content and with the methacrylic acid content of the interpolymer. Homogeneous gels were obtained when the polymer content ranged from 2.5 to 9%, and when the methacrylic acid content of the interpolymer was varied between 0.05 and 1.0%. To produce strong resilient gels a methacrylic acid concentration in the interpolymer of 0.1 to 0.3% appears essential, while greater concentrations make solution of the polymer in gasoline more difficult and impart to the gels a tendency to separate during storage at room temperature. In -40°C. surveillance tests the best caustic-interpolymer-gasoline gels (without toluene) developed more than 20% syneresis in three weeks. In 24-hour tests at -50°F. on a series of gels containing 5% interpolymer, 1% of 40% NaOH and 94% gasoline the syneresis observed increased regularly from 4 to 13% as the methacrylic acid content of the interpolymer was increased from 0.1 to 1.0%.

B. Caustic Strength and Concentration

In formulation studies on the use of NaOH as gelation agent, the strength of the caustic solution is apparently the major factor, since homogeneous gels were only obtained when the concentration of the caustic solution was greater than 10%. A gel which contained 5% of 15% caustic solution was stiff but was somewhat friable. Strong, only slightly fluid gels with no tendency towards friability resulted from the use of 40% aqueous caustic. To neutralize the acidity of the interpolymer requires less than 0.1% of 40% caustic. Strong stiff gels, however, are only obtained when the concentration of 40% caustic solution exceeds 0.5%. Best results were obtained with 1% of 40% NaOH as the gelation agent.

C. Gelling Agents other than NaOH

Apparently any strong base can be substituted for NaOH without material change in the physical properties of the gel, and without improvement in gel stability. Gelation was delayed 10-15 minutes by the use of calcium or barium hydroxide slurries. Other strong bases (Li, K) promoted gelation within one minute, while weak bases such as ZnO, $Al(OH)_3$ and Na_2CO_3 caused no gelation in 24 hours.

D. Detergents

The Eastman Kodak Company (contract OEMsr-538, Monthly Progress Report #5, October 14, 1942) reported that syneresis at -40°F. for one month could be reduced to 11-14% by the addition of 0.1 to 1.0% of certain oil in water emulsifiers. This was confirmed in tests in this laboratory on "GD-238", a product of the Organic Chemicals Department of E. I. du Pont de Nemours & Company, and "Sorbitan Monolaurate" produced by the Atlas Powder Company. The gels containing these wetting agents were exceptionally stiff and tough. When relative stability was obtained at -40°, however, separation in surveillance tests at 125°F. reached 20% after one week.

E. Cosolvents

Of the cosolvents tested only toluene improved gel stability at low temperature. Benzene tended to separate and freeze out of gasoline gels, while oxygenated solvents were without effect. These latter cosolvents, moreover, yielded gels which were unstable at 125°F. Interpolymers containing 2 or 3% methacrylic acid were solubilized in gasoline by the addition of 0.4% acetic acid, but the resulting gels were not promising.

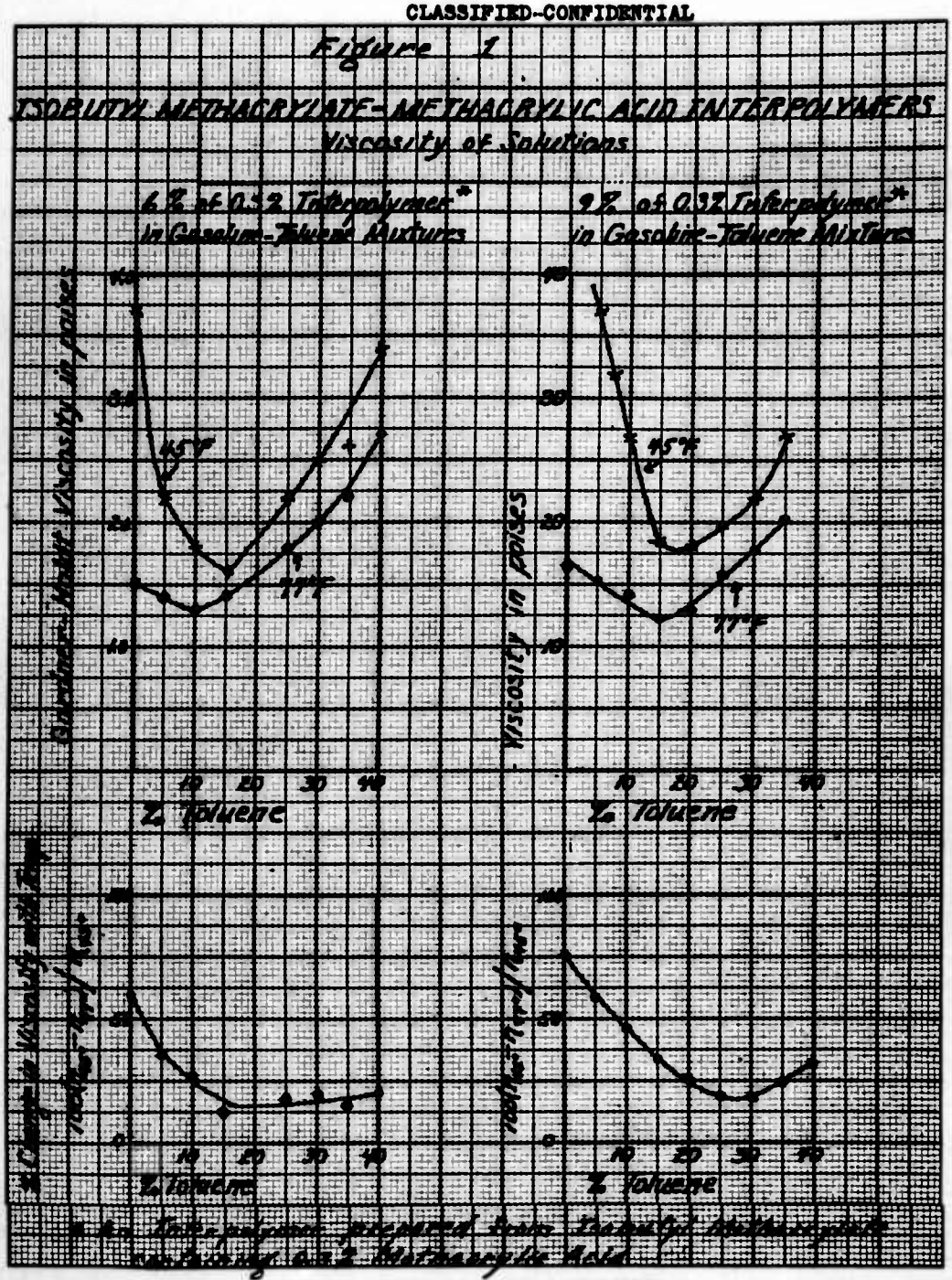
A study of the viscosity of solutions of interpolymer containing 0.3% methacrylic acid in gasoline-toluene mixtures indicated that at 45°F. in the range of 15 to 20% toluene, the viscosity approached a minimum and most nearly approached the viscosity of the same solution at 77°F. (see Fig. 1).

The stability of gels prepared by the addition of 1% of 40% NaOH to the above solutions again indicated that a toluene content of approximately 20% was unique. Gels containing 40% toluene were fluid and stratified after one week at room temperature, while some room temperature and low temperature syneresis was apparent when the toluene content was 15%. Not only surveillance but also physical properties favor the use of 20% toluene in gasoline. The addition of toluene imparts fluidity softness and lack of friability without a sacrifice of gel

Figure 1

ISOBUTYL METHACRYLATE-METHACRYLIC ACID INTERPOLYMERS

Viscosity of Solutions



* Interpolymers prepared from Isobutyl Methacrylate and Methacrylic Acid

strength. Thus the resistance to flow at low shearing forces, as measured by the parallel plate method, is reduced, while the resistance to rupture under high shearing forces, as measured by the Impact Strength, is slightly enhanced. For example, gels prepared from 4% of an interpolymer containing 0.3% methacrylic acid and 1% of 40% NaOH have the following properties:

15% toluene; Parallel Plate 6.7 cm. Impact Strength 14.6 units
20% " ; " " 7.7 " (softer) " 14.8 " (stronger)

The few gels prepared with more than 6% interpolymer showed no increase in impact strength with polymer content. The high polymer gels were somewhat friable and exhibited syneresis at room temperature. It is believed that a toluene content in excess of 20% would improve the properties of these gels.

F. Bodying Agents

Small amounts of soaps and inert fillers were added to the best interpolymer caustic gels in an attempt to reduce their resilience and to extend the polymer. The effect of these agents is demonstrated in Table 2 in two series of gels of constant polymer content. In accordance with the recommendations of formulation studies on high soap content gels, one and a half equivalents of 40% NaOH, in addition to the 1% of 40% NaOH standard for interpolymer gels, were added for each equivalent of acidic bodying agent. Naphthenic acid apparently acts as a plasticizing agent, effecting a marked increase in parallel plate value and decrease in the modified mobilometer value. Despite the increased fluidity and softness of naphthenic acid-containing gels, the impact strength was high. The presence of naphthenic acid therefore reduces resilience and improves adhesion without simultaneously reducing gel strength. The addition of stearic acid or Turkey red oil (sulfonated castor oil) stiffened and strengthened interpolymer gels but seriously reduced the burning rate. The resultant gels are similar to AE polymer gels of higher soap content rather than interpolymer gels. Gels containing alpha cellulose, which acts as an inert filler, were not resilient and had the lowest parallel plate value in each series. In surveillance tests at 125°F. alpha cellulose slowly settled out of the stable interpolymer gels.

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Table 2

Effect of Bodying Agents on Gel Properties

A. Gels containing 4% Interpolymer (0.3% methacrylic acid)

Bodying Agent	Impact Strength (C Scale)	Modified Mobilometer (min/1"/54g)	Parallel Plate Value (cm.)	Relative Burning Times (min.)
None	14.8	0.08	7.7	2.8
1% stearic acid	17.4	0.14	7.4	3.3
1% naphthenic acid	17.1	0.01	10.7	2.7
1% T-132 resin	14.4		7.1	
0.5% alpha cellulose	16.8	0.66	6.8	4.3

B. Gels containing 3% Interpolymer (0.3% methacrylic acid)

Bodying Agent	Impact Strength (C Scale)	Modified Mobilometer (min/1"/54g)	Parallel Plate Value (cm.)
0.5% stearic acid	11.0	0.3	8.2
0.5% naphthenic acid	10.0	0.1	10.1
0.5% T.R.O.	11.5	0.11	8.0
0.5% alpha cellulose	11.0	0.16	8.0

G. Gasoline

The composition of the gasoline markedly affects the strength and stability of interpolymer gels. Throughout this study a special naphtha from the Pure Oil Company meeting CWS specification 196-131-124 was used as the fuel component. Gel A-3489 (4% interpolymer prepared from isobutyl methacrylate containing 0.3% methacrylic acid, 1% of 40% NaOH, 20% toluene, 75% gasoline) was also prepared in five other grades of gasoline. As shown in Table 3 the syneresis observed when the gels were held at -40°F. was directly dependent on the aniline point of the gasoline. On this basis a fuel with an aniline point of 120°F. is not acceptable. That aniline point alone is not a sufficient specification is shown by the fact that gelation did not occur in Gulf No Nox gasoline (aniline point 106°) but did occur in gasolines with both higher and lower aniline points. The gel prepared in Blue Sunoco was stiffer and considerably stronger (impact strength 13.5 D units) than that prepared in Purol

Table 3

Effect of Gasoline Quality on A-3489 Gels

Manufacturer	Gasoline Trade Name	Date Procured	Aniline Point	Octane Number	Parallel Plate cm.	% Syneresis after 1 week at -40°F.	Time Required for Gelation at 25°F.
Pure Oil Co.	Naphtha #2	12-1-43	95	80		0	5 min.
Sun Oil Co.	Blue Sunoco	2-1-44	95			0	1/2 "
Gulf Oil Co.	No Nox	4-1-42	106	79	No gelation		
Tide Water Oil Co.	Tydol Ethyl	4-1-42	111.5	80	6.2	0.2	5 "
Cities Service Oil Co.	Koolmotor winter	4-1-42	111.5	70	6.6	0.8	1-3 hours
Cities Service Oil Co.	Koolmotor summer	4-1-42	120	70	6.5	7.0	" "

A-3489: 4% Interpolymer from isobutyl methacrylate containing 0.3% methacrylic acid.
1% of 40% NaOH.
20% Toluene.
75% Gasoline.

gasoline (gel impact strength 11.5 D units) with the same aniline points, 95°F. The rate of gelation at 25°F. divides the gasolines tested into four classes: (a) Blue Sunoco - gel formed less than one minute after addition of caustic; (b) Purol, Tydol Ethyl - mixture sets to a gel in one to ten minutes; (c) Cities Service Koolmotor, winter and summer grades - gelation is gradual, and to obtain a uniform mixture stirring must be continued for up to three hours after mixing; (d) Gulf No Nox - no gel forms in 24 hours.

The effect of toluene content on gel properties described in Section E suggests that the aromatic content of the fuel may be an important variable. Such information is not available on the fuels tested. The above data indicate that the suitability of a gasoline for use in preparing interpolymer gels must be determined by laboratory test. It is recommended that further work on gasoline specifications be undertaken.

V. Firing Tests

Interpolymer caustic gels have been tested as incendiary fillings for both the M-47 and the E-9 bombs. The tests in M-47 bomb were made before the firing index system was developed, so that quantitative data on this munition are not available. The observations on the drop tests run at Edgewood Arsenal June 8 and 16, 1942, recorded in Table 4, are the consensus of opinion of the CWS personnel present. It was unanimously agreed that the gel prepared from 5% of an interpolymer containing 0.1% methacrylic acid (Test 1) had firing characteristics superior to any other gel tested, including the standard IM-1 filling. The interpolymer gel was distributed in larger pieces of more uniform size and burned more intensely than did the IM-1 gel. During combustion the interpolymer gels did not develop the heavy crust typical of soap containing gels.

To simulate the action of the 40-lb. ejection E-9 bomb, the Texas Company has developed a mortar of similar size and shape actuated by the proper explosive charge and functioning in an identical manner (see Texas Company N.D.R.C. contract 11-422 monthly report for November 15, 1943). Twenty methacrylate gels were submitted for evaluation by the Texas Company in this test, of which six gels contained interpolymer and only 1% or less of soap-forming acids. The composition and physical properties of these six gels are given in the appended Summary Sheet, Table 5, and the results of the firing tests in Summary Sheet, Table 6. Possibly due to slow ejection and to a shift of the mortar during recoil, aiming of the mortar for methacrylate gels was inadequate. The gels always hit the target near

Table 4

Firing Tests at Edgewood Arsenal June 8 and 16, 1942

Gels loaded in M-47A1 bombs with M-13 bursters dropped from 5,000 feet onto factory structure.

	Gel Composition				
Test	% Interpolymer	% Methacrylic Acid in Interpolymer	% 40% NaOH	Bombs Tested	Results
1	5	0.1	1	4	Fastest burning gel and largest particle size
2	5	0.3	1	3	Nearly equivalent to Test 1
3	5	0.6	1	1	Equal to Test 6 and 7 but inferior to 1
4	3.5	0.4	1	1	Excessive flash. Gel particles too small
5	3.5	0.6	1	1	
6	IM-1 gel			3	Particle size smaller and flash greater than in Test 1
7	F-107 gel			3	

the top, in general left of the center, and a large portion of the material frequently went over the top of the target.

It was, however, possible to observe the action of the gel on impact, the tendency of the gel to flow during combustion and the rate at which large quantities of gel ignited the plywood target. All four gels containing 4% interpolymer on impact flowed down the vertical target and remained on the floor in contact with the back wall. The two gels containing more than 4% interpolymer were apparently too resilient, since they tended to bounce from the target. When at least two pounds (20% of the total charge) of the gel was near combustible material, the fire burned through the plywood target in from 1 to 1-1/2 minutes. Once the plywood was ignited, a very intense fire broke out, reaching a peak at 2 to 4 minutes after the gel reached the target. Within 5 minutes all combustible material in the immediate vicinity had been consumed. Largely due to its central location, gel A-3942, containing 6% interpolymer, gave the greatest target destruction in 10 minutes, namely 70%. When at least 35% of the 4% interpolymer gels remained in the target area, the target destruction was 40-55%. The gel containing naphthenic acid was the most adhesive and exhibited the most flow during combustion. The latter property should assist lateral spread of a fire on a larger target. A tendency for the gel to spread during the fire was observed with the straight 4% interpolymer gel and also the interpolymer gel bodied with alpha cellulose, but not with the gel containing stearic acid nor the straight 6% interpolymer gel. Based on ease of manufacture, rapid burning rate and successful destruction of the Texas target, the gel prepared from 4% interpolymer containing 0.3% methacrylic acid has been chosen for more exhaustive evaluation in the E-9 bomb.

Report prepared by E C Kirkpatrick
E. C. Kirkpatrick

Approved by John S. Beekley
John S. Beekley,
Official Investigator

VI. Appendix - Summary Charts

The composition, physical properties, and surveillance of all interpolymer gels are shown in Summary Chart, Table 5. The field data on E-9 mortar tests at the Texas Company laboratories are given in Summary Chart, Table 6.

The source of the materials used in gel preparation was as follows:

Interpolymer: Various interpolymers of isobutyl methacrylate and methacrylic acid (abbreviated as M.A.) produced by the Ammonia Department, E. I. du Pont de Nemours & Company.

Stearic Acid: Hydrofol 51 from the Werner G. Smith Company.

Naphthenic Acid: Nuodex Products Company Naphthenic Acid 240.

T-132: T-132 resin from Stanco Distributors.

T.R.O.: Turkey Red Oil from Hercules Powder Company.

CaO: USP grade containing more than 95% CaO. To obtain uniform size it was ground, screened, and composited to

35-60 mesh	22%
60-80 "	22%
80-100 "	22%
100-200 "	22%
pass 200 "	12%.

Toluene: Toluol (commercial grade).

Gasoline: To meet C.W.S. Specification 196-131-124 from Pure Oil Company.

Preparation of the gels involves adding with strong agitation caustic to a gasoline stock solution containing polymer and soap forming acids. In preparing the stock solution, it is preferable to dissolve the soap forming acids in the gasoline before adding the methacrylate polymer. The polymer should all be added with rapid agitation during a one-minute period to the gasoline, which is at a temperature above 20°F. Stirring should be continued until solution is complete.

The technique involved in determining the physical properties of gels has been described in a memorandum by Kirkpatrick on IM-3 gels which was circulated May 17, 1943 (letter Strain to General W. C. Kabrich) and will be discussed

in more general terms in our final report. The impact strength measures in empirical units the energy required to force a rod through a sample of gel. When the value is marked by an asterisk, the rod did not swing completely free of the gel, so that only a minimum, not a true value, was obtained. The modified Mobilometer test involves the determination of the rate of fall of a Gardner 4-hole Mobilometer plunger through a sample of gel in a one-quart bottle. The parallel plate value is the diameter of the circle to which 5 cc. of gel will spread in one minute between parallel glass plates under a 2-kilogram load. All of these tests determine gel consistency at various rates of shear.

The notebooks in which the original data are recorded have been deposited in the Ammonia Department Files and are available for reference. These are N.B. 884, 965, 973, 998, 1049, 1118, 1152.

TABLE 5

SUMMARY SHEET

[illegible]

COMPOSITION AND PROPERTIES OF ISOBUTYL METHACRYLATE-METHACRYLIC ACID INTERPOLYMER GELS

GEL	DATA BOOK	PAGE	Interpolymer %	Interpolymer % M.A.	Composition Base %	Composition Base Name	Miscellaneous %	Miscellaneous Name	40% NaOH %	Physical Properties: Impact C Scale	Physical Properties: [illegible] D Scale	Physical Properties: [illegible]	Physical Properties: Plate Diam. [illegible]	Surveillance -40° F. Days	Surveillance -40° F. Days	Surveillance 125° F. Days	Surveillance 125° F. Days	Remarks
			40 % NaOH															
379	884	99	2.5	2.0					1.0									Polymer did not dissolve.
[illegible]	"	"	2.5	1.0					1.0					12	2			Very fluid, weak, very elastic.
[illegible]	"	"	2.5	1.0					1.0					10	2			" " ,formed 2 layers ageing.
[illegible]	"	"	2.5	1.0					1.0					[illegible]	2			" " ,weak, very elastic.
[illegible]	"	100	2.5	1.0					1.0					7	2			Same as above. [illegible] stored 1 yr.
[illegible]	"	99	5.0	1.0					1.0					11	2	0	30	Fairly stiff, very strong, very elast.
[illegible]	"	"	2.5	0.6					1.0					7	2			Slightly fluid, weak.
[illegible]	"	"	2.5	0.6					1.0									Same as above. [illegible] stored 1 yr.
[illegible]	"	100	2.5	0.6					1.0									Same as above.
[illegible]	"	99	5.0	0.6					1.0					11	2	0	30	Very stiff, very strong, very elastic.
[illegible]	"	"	5.0	0.6					1.0									" " " "
[illegible]	"	"	2.5	0.6					1.0					8	2			Slightly fluid, strong.
[illegible]	"	"	2.5	0.4					1.0									" " ,weak.
316	"	98	5.0	0.4					1.0					5	3			Stiff, strong.
[illegible]	"	103	5.0	0.4					1.0					20	20			" "
[illegible]	"	112	5.0	0.4					1.0					20	13	0	30	" " [illegible] 1 yr.
2091	973	23	2.0	0.3					1.0									Fluid, strong, elastic. 90% [illegible] stored
2092	"	"	3.0	0.3					1.0									" very "
390	884	110	5.0	0.3					1.0					20	4			Sl. " "
388	"	113	5.0	0.3					1.0					20	20	0	30	" "
415	"	125	5.0	0.3					1.0									Sl. " "
[illegible]	"	99	5.0	0.3					1.0					8	2			" "
2093	973	23	3.0	0.1					1.0									" " 90%
2094	"	"	4.0	0.1					1.0									" " 90%
380	884	112	5.0	0.1					1.0					20	12	0	30	" weak
387A	"	114	5.0	0.1					1.0					20	13	0	30	" strong.
[illegible]	"	99	5.0	0.1					1.0									Stiff.
349	"	"	5.0	0.1					1.0									"
[illegible]	"	104	5.0	0.1					1.0									Fluid, weak
[illegible]	"	106	5.0	0.1					1.0									Fluid, strong.
396	"	125	5.0	0.1					1.0									
			CAUSTIC STRENGTH AND CONCENTRATION															
314	884	98	5	0.4					5.0					6	3			314-329 all formed gels. 316 gave about the optimum in properties. Higher NaOH concentration and amount produce increased stiffness and strength. The concentration of the NaOH solution used has a greater effect than the percent of NaOH solution used.
315	"	"	5	0.4					2.5					[illegible]	3			
316	"	"	5	0.4					1.0					[illegible]	3			
317	"	"	5	0.4					0.5					[illegible]	3			
318	"	"	5	0.4			5.0	25% NaOH						[illegible]	3			
319	"	"	5	0.4			2.5	"						[illegible]	3			
[illegible]	"	"	5	0.4			1.0	"						[illegible]	3			
[illegible]	"	"	5	0.4			0.5	"						[illegible]	3			
[illegible]	"	"	5	0.4			5.0	15%						[illegible]	3			
[illegible]	"	"	5	0.4			2.5	"						[illegible]	3			
[illegible]	"	"	5	0.4			1.0	"						[illegible]	3			
[illegible]	"	"	5	0.4			0.5	"						[illegible]	3			
[illegible]	"	"	5	0.4			5.0	5%						[illegible]	3			
[illegible]	"	"	5	0.4			2.5	"						[illegible]	3			
[illegible]	"	"	5	0.4			1.0	"						10	3			
329	"	"	5	0.4			0.5	"						[illegible]	3			
[illegible]	"	80	2.5	1.0			2.5	10%						5	3			2 layers formed.
[illegible]	"	[illegible]	2.5	0.5			2.5	"										Soft, fairly strong, elastic.
[illegible]	1118	[illegible]	3	0.3			0.28	30%										3099, 3100, 3102 were good gels, and increasing the amount of caustic increased the strength of the gel.
3099	"	[illegible]	3	0.3			0.56	"										
3100	"	[illegible]	3	0.3			[illegible]	"										
3102	"	[illegible]	3	0.3			1.36	[illegible]										
			ACETIC ACID AS POLYMER SOLUBILIZING AGENT															
[illegible]	884	91	2.25	1.0	0.4	Acetic	2.5	15% NaOH										Gel separated into 2 layers.
[illegible]	"	"	[illegible]	[illegible]	[illegible]	"	[illegible]	"										Soft, [illegible], 2 layers formed.
[illegible]	"	81	[illegible]	[illegible]	[illegible]	"	[illegible]	"										Soft, fairly strong, elastic.
[illegible]	"	[illegible]	[illegible]	[illegible]	[illegible]	"	[illegible]	"										Stiff, [illegible], elastic. [illegible] stored 1 yr.
[illegible]	"	[illegible]	[illegible]	[illegible]	[illegible]	"	[illegible]	"										Stiff [illegible], no elasticity.
[illegible]	"	[illegible]	[illegible]	[illegible]	[illegible]	"	[illegible]	[illegible]										Stiff, strong, elastic. [illegible]
[illegible]	"	91	[illegible]	[illegible]	[illegible]	"	[illegible]	[illegible]										" " "

[illegible]

TABLE 5

SUMMARY SHEET . (Continued)

[redacted]

COMPOSITION AND PROPERTIES OF ISOBUTYL METHACRYLATE-METHACRYLIC ACID INTERPOLYMER GELS

GEL	DATA BOOK	PAGE	Interpolymer %	Interpolymer % M.A.	Composition (Other) %	Composition (Other) Name	Miscellaneous %	Miscellaneous Name	40% NaOH %	Physical Properties: Impact Strength "C" Scale	Physical Properties: "D" Scale	Physical Properties: min./wt.		Physical Properties: Plate Diam, cm	Surveillance -40°F. Syn.	Surveillance -40°F. Days	Surveillance 125°F. Syn.	Surveillance 125°F. Days	REMARKS
OTHER GELLING AGENTS																			
369	884	104	5	0.1			1	50% Ca(OH)$_2$ slurry							20	9	20	25	
413		120	5	0.1	1.3	Water	2	CaO							20	14	0	30	
2004	973	2	5	0.1	1.3	"	1	LiOH							20	18	0	30	
[illegible]	884	138	5	0.1	0.3	"	0.4	Al(OH)$_3$											No gellation occurred.
[illegible]	"		5	0.1	1.3		2.3	[illegible]											No gellation occurred.
[illegible]	"	135	5	0.1	1.0	Stearic	2.0	CaO											Very stiff, strong, elastic.
122	"		5	0.6	1.0		2.0	CaO											"
370	"	104	5	0.1			1	50% Na$_2$CO$_3$ Slurry											No gelation occurred.
371	"		5	0.1			1	50% NaHCO$_3$ Slurry											No gelation occurred. 1 year
372	"	"	5	0.1			1	50% Ba(OH)$_2$ Slurry											Good gel but 10% syneresis stored/
COSOLVENTS AND BODYING AGENTS																			
1946	965	192	4	0.3			15	Toluene	1.0	14.6		.02	117g.	6.7					
1947	"	"	5	0.3			15	"	1.0	16.4		.04	"	6.4					
1948	"	195	6	0.3			15	"	1.0	17.4		.06	"	6.2					
1946A	"	"	4	0.3			15	"	1.0	10.0									4% Syn. after 2 weeks at 77°F.
1947A	"		5	0.3			15	"	1.0	9.3									" " " "
1948A	"		6	0.3			20	"	1.0	9.9									" " " "
[illegible]	1118	72	[illegible]	0.3			20	"	1.0	14.8		.08	5kg.	7.7	0	30	0	30	
[illegible]		68	[illegible]	0.3			20	"	1.0		13.7	.01	267g.	7.4	0	30	0	30	
[illegible]	884	112	5	0.1			10	Benzene	1.0						20	12	2	30	
[illegible]			5	0.1			10	"	1.0						20	12	0	30	
[illegible]	965	5	5	0.1			5	n-Butanol	1.0						20	13	0	30	
[illegible]	"	"	5	0.1			5	Hexanone	1.0						20	12	20	5	
[illegible]	"		5	0.1			5	dibutylether	1.0						20	12	20	7	
[illegible]	1118	65	[illegible]	0.3	1	T-132	15	Toluene	1.5	14.4		.01	267g.	7.1	20	12	20	5	
[illegible]	"	"	[illegible]	0.3	1	Stearic	15	"	1.5	16.2		.01	267g.	7.1					
[illegible]	"	"	[illegible]	0.3	1	Naphthenic	15	"	1.5	14.0		.01	267g.	10.8					
[illegible]	"	"	[illegible]	0.3	1	T.I.O.	15	"	1.5	16.2		.20	267g.	7.1					
[illegible]	"	72	[illegible]	0.2	1	Stearic	20	"	1.4	16.7		.01	5kg.	10.0	0	30	0.1	30	
[illegible]	"	"	[illegible]	0.2	0.5	a-Cellulose	20	"	1.0	13.0		.12	5kg.	8.5	0	30	0	30	
[illegible]	"	71	[illegible]	0.3	0.5	Stearic	20	"	1.0	8.3		.01	5kg.	9.1					
[illegible]	"	"	[illegible]	0.3	0.5	"	20	"	1.0	11.0		.03	5kg.	8.2					
[illegible]	"	72	[illegible]	0.3	1	"	20	"	1.2	17.3		.14	5kg.	7.1					
[illegible]	"	68	[illegible]	0.3	1	"	20	"	1.0	16.9	12.3	.03	267g.						
[illegible]	1152	5	6	0.3	0.5	"	20	"	1.5		11.9			6.6					
[illegible]	1118	71	[illegible]	0.3	0.5	Naphthenic	20	"	1.1	5.9		.01	5kg.	12.1					
[illegible]	"	"	[illegible]	0.3	0.5	"	20	"	1.1	10.0		.01	5kg.	10.1					
[illegible]	"	"	[illegible]	0.3	1.0	"	20	"	1.5	17.1		.01	5kg.	10.7	0.8	30	0.4	30	
[illegible]	"	"	1.5	0.3	0.1	T.I.O.	20	"	0.8	4.9		.01	5kg.	10.2					
[illegible]	"	"	2	0.3	0.5	"	20	"	1.3	7.0		.02	5kg.	8.6					
[illegible]	"	"	3	0.3	0.5	"	20	"	1.3	11.5		.11	5kg.	8.0					
[illegible]	"	"	3	0.3	0.5	a-Cellulose	20	"	1.0	11.0		.16	5kg.	8.0					
[illegible]	"	72	4	0.3	0.5	"	20	"	1.0	16.8		.66	5kg.	6.8	0	30	0	30	Cellulose settled out at 125°F.
[illegible]	"	"	4	0.3	1	"	20	"	1.0			.19	5kg.						Poor a-cellulose dispersion
DETERGENTS																			
767	958	48	5	0.1	0.1	GD 278 (du Pont)			1.0					8.4	12	30	20	7	Stiff, strong, very elastic
[illegible]	"	"	5	0.1	1.0	GD 278 (du Pont)			1.0					9.4	13	30	20	7	" " " "
[illegible]	"	"	5	0.1	0.1	Sorbitan monolaurate (Atlas)			1.0					7.8	14	30	20	7	" " " "
770			5	0.1	1.0	Sorbitan monolaurate (Atlas)			1.0					5.8	20	10	20	21	V. " " "
TEXAS CO. BOMB (E-9) SHIPMENTS																			
[redacted]	1118	73	4	0.3			20	Toluene	1.0	15.2	10.2	.01	117g.	7.5	0	30	0	30	
[redacted]	"	[illegible]	6	0.3			20	"	1.0		13.7	.06	117g.	6.8	0	30	0	30	
[redacted]	"	75	4	0.2	1	Stearic	20	"	1.1	16.6	11.7	.02	117g.	8.0	0	30	0.1	30	
[redacted]	"	"	5	0.2	0.25		20	"	1.3		13.8	.14	117g.	6.8	0	30	0.2	30	0.5% a-Cellulose also.
[redacted]	"	"	4	0.3	1	Naphthenic	20	"	1.3	11.5	15.2	.01	117g.	10.0	0.8	30	0.4	30	
[redacted]	"	"	4	0.3	0.5	a-Cellulose	20	"	1.0	15.3	11.1	.33	117g.	6.2	0	30	0	30	a-Cellulose settled out at 125°F.

[redacted]

TABLE 6

SUMMARY SHEET

INCENDIARY EVALUATION OF INTERPOLYMER GELS BY THE TEXAS COMPANY AT BEACON, NEW YORK, NOVEMBER 12 AND 26, 1943

SAMPLE	• COMPOSITION Interpolymer %	Interpolymer % T.E.A.	% Stearic Acid	% Naphthenic Acid	% Alpha Cellulose	% of 80% Base	LOCATION OF INCENDIARY MATERIAL	DESCRIPTION OF FIRE	% of Gel Burned	% of Gel in Effective Location	% of Target Destroyed in 10 min.
A-3489	4	0.5				1	20% beyond target. 80% in left corner at base of target. Gel flowed out 5' x 2-1/2' area at 2 min.	Entire left half of target burning in 1 min. Intense fire with flames reaching 5' above target at 2-3 min. At 4 min. left side of target consumed. A slow wood fire at edge of burned area remained.	80% in 4 min.	80	55
A-3489 repeat							10% beyond target. 90% in left corner at base of target. Gel flowed over flame to maximum area of 4' x 3', then flowed off apron. Gel out of target area at 5 min.	Fire burned through plywood in 1-1/2 min. Intense fire at 3 min. Left side of target destroyed in 5 min.	80% in 10 min.	90	45
A-3487	4	0.5		1		1.5	80% beyond target. 5% in gun. 15% adhered to vertical wall.	Flash fire charred surface of 30% of target in first min. Very low fire thereafter.		15	0
A-3487 repeat							60% beyond target. 5% in gun. 35% in right corner base of target which spread in 1 min. over 4' x 2' area of floor.	Fire burned through wood in 1 min. Peak of fire at 3 min. Gel and right side of target consumed in 5 min.	90% in 5 min.	95	45
A-3488	4	0.5			0.5	1	5% scatter. 95% in left corner base of target, covering 2' x 3' area on floor.	Flames reached top of target in 1 min. and 20% of target was burning at 2 min. Wood near gel destroyed at 5 min. A slow wood fire unsupported by gel continued during 5-10 min. period.	95% in 10 min.	95	45
A-3488 repeat							Entire charge in 1 piece hit top edge of target. 70% carried beyond the target. Tail came and 10% gel fell to ground. 20% of gel reached base of target.	Fire burned through wood in 2 min. A steady fire burned out a 2-1/2' strip from top to bottom of the target.	90% in 10 min.	20	11
A-3490	4	0.2	1.0			1.4	85% beyond and to left of target. 10% at base of target in center, covering 1' x 2-1/2' area. 5% in left corner at base of target.	A slow steady local fire burned through wood in 5 min.		15	11
A-3491	5	0.2	0.25		0.5	1.2	70% hit wall and bounced to ground. 10% on apron. 10% in gun. 10% beyond target.	Fire put out at 5 min. after the gel had ignited the wooden floor.			
A-3942	6	0.5				1	95% on apron 1 foot from vertical wall. 5% on ground.	Fire burned through plywood in 1-1/2 min. Intense fire during 2-3 min. period. 60% of target destroyed in 6 min. An unsupported wood fire continued during 6-10 min. period.	100% in 10 min.	95	70

TEST TECHNIQUE: Approximately 10 pounds of gel placed in E-9 mortar and fired at cinder block backed plywood target. Fire extinguished at 10 min.

• Composition: All gels contained 20% talmoen, polymer comps and caustic as indicated above, and the remainder (75-75%) Furol gasoline of 95°F. aniline point.

CONFIDENTIAL

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ABSTRACT:

The development of stable isobutyl methacrylate-methacrylic acid interpolymer thickened gasoline gels is reviewed. Laboratory formulation studies on the effect of the nature and the concentration of the polymer, the gelation agent, the soap forming acids, and the fuel composition are described. The evaluation of representative gels as the fillings for large incendiaries are discussed. A soap free gel containing from 3 to 6% of an interpolymer prepared from isobutyl methacrylate with 0.3% methacrylic acid, 20% toluene, 1% of 40% aqueous NaOH, and from 76 to 73% gasoline gave best results in firing tests in the M-47 and E-9 incendiary bombs. Such a gel has high strength, a rapid burning rate, and a high fuel content.

NW 22
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Incendiary Bombs