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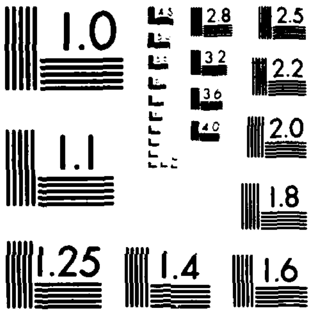

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IMPROVED ACRYLIC SYSTEMS FOR RAPID RUNWAY REPAIR

U.S. NANDI, R.G. SINCLAIR, P.C. BEHRER,
J.P. BOYER AND M.J. SNYDER

BATTELLE'S COLUMBUS LABORATORIES
505 KING AVENUE
COLUMBUS, OHIO 43201

MAY 1983

FINAL REPORT
JUNE - SEPTEMBER, 1982

ENGINEERING & SERVICES LABORATORY
AIR FORCE ENGINEERING & SERVICES CENTER
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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER
ESL-TR-82-46

2. GOVT ACCESSION NO.
AD-A130389

3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)
Improved Acrylic Systems for Rapid Runway Repair

5. TYPE OF REPORT & PERIOD COVERED
FINAL
June-September, 1982

6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)
U. S. Nandi, R. G. Sinclair, P. C. Behrer, J. P. Boyer and M. J. Snyder

8. CONTRACT OR GRANT NUMBER(s)
F08635-82-C-0315

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Battelle's Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS

11. CONTROLLING OFFICE NAME AND ADDRESS
Air Force Engineering and Services Center
Tyndall Air Force Base, Florida 32403

12. REPORT DATE
May 1983

13. NUMBER OF PAGES
30

14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)

15. SECURITY CLASS. (of this report)
Unclassified

15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)
Approved for Public Release; Distribution Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES
Availability of this report is specified on reverse of front cover.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Rapid Runway Repair
Bomb Damage Repair
Polymer Concrete
Acrylic Polymer Research
Material Development
Pavement Repair
Fast-Setting Concrete

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The objectives of this research were to achieve a basic improvement in the water compatibility of acrylic materials and to utilize lower cost acrylic materials, while maintaining the desirable characteristics of the DOMA system: rapid strength development over a full environmental range, high strength and toughness, low viscosity, minimum flammability and toxicity (con't)

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Unclassified

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hazards, long shelf life, and ease of handling. The resultant HEMA resin yielded about 100-percent wet strength improvement with cost lowered to approximately $1.13 per pound from $2.17 per pound for DOMA These improvements led to inclusion of HEMA in the Advanced Bomb Damage Repair Systems subtask of the Rapid Runway Repair Program.

PREFACE

This report was prepared by the Columbus Laboratories of the Battelle Memorial Institute, Columbus, Ohio, under Contract No. F08635-82-C-0315, for the Air Force Engineering and Services Center, Engineering and Services Laboratory (AFESC/RD), Tyndall AFB, FL 32403.

This report summarizes work done between 1 June 1982 and 30 September 1982. Captain Daniel J. Pierre, was the AFESC/RDCR project officer.

This report has been reviewed by the Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication.

Daniel J. Pierre

DANIEL J. PIERRE, Capt., USAF
Project Officer, Bomb Damage Repair

JOHN E. GOIN, Lt. Col., USAF
Chief, Engineering Research Div.

Francis B Crowley III

FRANCIS B. CROWLEY III, Col., USAF
Director, Engineering and Services
Laboratory

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SECTION I
INTRODUCTION

1. BACKGROUND

The rapid repair of bomb-damaged runways after an enemy attack is important to the Air Force. One means of accomplishing this repair is to backfill the bomb crater with rock aggregate and form a strong structural cap at the top. This cap would consist of aggregate and cured monomers and resins. The aggregate and resin might be premixed for placement or (as in the research reported herein) the cap might be formed by percolation of liquid resin through the porosity of loosely placed aggregate. Figure 1 illustrates such a system. Acrylic-based cap materials were suggested and developed under a previous program on the Advanced Materials Application research of the AFESC's Rapid Runway Repair Program (Reference 1). The ability to repair pavements under a variety of ambient conditions makes the use of acrylic-based cap materials an excellent prospect for the rapid repair of bomb-damaged runways. Contrary to standard concrete, one of the primary advantages of these materials is the short time required to cure them and reach high strength. In the previous program it was demonstrated that an acrylic system based on dicyclopentenyl oxyethyl methacrylate (DOMA) met most of the goals of an acrylic cap system. It cured rapidly over a wide temperature range and possessed no significant health or flammability hazards during use. However, the cost of DOMA increased from $1.52 to $2.17 per pound during 1981. This report discusses the development of an acrylic system with improved material costs without significant penalties in property performance. In addition, improvements were sought in the water compatibility of the acrylic system, and some of the more fundamental aspects of the new acrylic system were studied. This development was begun in June 1982 and completed in September 1982, with substantial improvements over the original DOMA system.

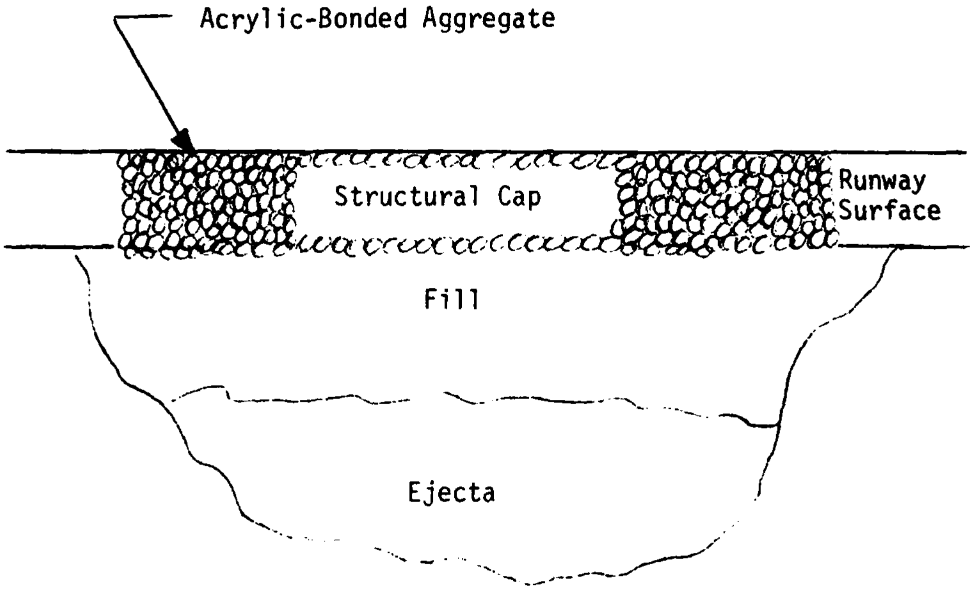

Figure 1. Generalized Free-Form Crater Repair

2. OBJECTIVE

The main objective of this research program was the initial development of an alternate acrylic curing system which will offer substantial advantages over the DOMA acrylic system developed for AFESC by Battelle on the BDM/BCL Task Order Contract. The goals were to achieve a basic improvement in the water compatibility of acrylic materials and to utilize lower cost acrylic materials, while maintaining the desirable characteristics achieved with the DOMA system: rapid strength development over the full environmental range, high strength and toughness, low viscosity, minimum flammability and toxicity hazards, long shelf life, and ease of handling.

3. SUMMARY

The major objectives of improving water compatibility and reducing cost were achieved with the identification of hydroxyethyl methacrylate (HEMA) resin, using percolation-forming techniques. This variation of the basic DOMA resin was demonstrated to be capable of approximately 100 percent better wet strength and costs approximately $1.13 per pound as opposed to $2.17 per pound for DOMA. Based on the nature of the results with the HEMA resin, this resin was included in the Advanced Bomb Damage Repair (BDR) Systems subtask of the Rapid Runway Repair Program as a replacement for the DOMA resin.

In the final analysis, the most practical BDR system will be one with the most technical, cost, and safety advantages.

From this perspective of the BDR objective, a brief attempt was made to identify a suitable replacement for the dibenzoyl peroxide initiator, that is, one that was liquid and pumpable. Preliminary results with the new liquid initiator system were encouraging. The new initiator system was developed by dissolving the solid benzoyl peroxide in a mixture of dibutyl phthalate and dibutyl peroxide. Dibutyl phthalate is a plasticizer and, when added in small amounts, imparts slight reductions in the cure shrinkage and modulus of the cured system. This is advantageous since crazing during rapid curing is thereby avoided, in addition to providing a pumpable initiator component. Thus, the completed BDR system has one component that is comprised of monomers chosen because they:

a. Cure quickly over wide ambient conditions.

b. Adhere to wet aggregate.

c. Provide high strength when cured.

d. Have a low viscosity and mix well with the aggregate.

e. Have a density greater than water, which allows displacement of the water.

f. Possess very favorable safety factors.

g. Are a mix of commercially available materials with the lowest cost consistent with the above.

The second component of the BDR system is the accelerator, or "trigger ," which is added to the first component during placement. Mixing is anticipated as being performed in a simple "Y" valve that can meter the approximate amount of required initiator, depending upon ambient temperature.

SECTION II

APPROACH AND FINDINGS

1. SELECTION OF CANDIDATE MATERIALS

Selection of materials followed polymer chemistry logic as follows:

a. Seek fast curing to high-strength, liquid systems. This culled the list immediately to organic chemicals that include epoxies, styrenes, acrylates, methacrylates, and polyurethane precursors.

b. Choose least expensive materials that are free from toxicity and flammability problems. This removed epoxies, styrenes and some methacrylates, acrylates and urethanes.

c. Test those curable systems that promise good wet adhesion and fast curing at lower temperatures.

Some methacrylate and acrylate monomers were found to have all the above potential advantages, based on review of the technical literature and previous experience. That is, the acrylic monomers were liquids that would be easily pumpable and flowable by the percolation technique into the backfilled aggregate in the bomb crater. The monomers were cross-linkable into strong, fast-curing thermosets. Selection of materials to be tested focused on members of the acrylic class of monomers that were safe and offered good water compatibility. This selection could be aided by inspection of the chemical formula, e.g., its molecular weight, polarity, and hydrophilicity, as well as any listed data.

Technical literature and manufacturer's brochures and data sheets were reviewed (References 2-5) and discussions with other Battelle polymer chemists and vendors were carried out to select candidate materials. Commercially available materials, for which available data indicated improved water compatibility and lower cost than DOMA, without sacrificing other desirable properties, were taken into consideration.

Information gathered on several candidate acrylic prepolymers is shown in Table 1.

Hydroxy methacrylates were selected from this list for several reasons. They are a comparatively low-cost material, readily available

TABLE 1. SELECTION OF MATERIALS FOR ALTERNATIVE ACRYLIC SYSTEMS

Monomer	Flash Point, °F	Boiling Point, °F	Freezing Point, °F	Supplier	Price per Pound
Dicyclopentenyl Oxyethyl Methacrylate (DOMA)	>200	662	<-35	Rohm and Haas	$2.17
Hydroxy Ethyl Methacrylate (HEMA)	214	388	<-40	Rohm and Haas	1.05
Hydroxypropyl Methacrylate (HPMA)	208	406	<-40	Rohm and Haas	1.05
Ethyl Hexyl Acrylate (EHA)	188	416		Celanese	0.67
Butyl Acrylate (BA)	102	298		Celanese	0.57
Tetraethylene Glycol Diacrylate (TTEGDA)	>200			Celanese	1.35
Trimethylolpropane Trimethacrylate (TMPTMA)	>200			Celanese	1.80
Maleic Anhydride (MA)	Solid			Exxon, Dow, Monsanto	0.47
Acrylic Polyester				Virginia Chemical	1.49
Acrylic Epoxy				Celanese	1.63
Acrylic Acrylate				Celanese	--
Hexanediol Diacrylate (HDDA)	>200	>600		Celanese	1.35
Tripropylene Glycol Diacrylate (TRPGDA)	200	>600		Celanese	1.35
Pentaerythritol Triacrylate (PETA)		>600		Celanese	1.35

in the market, and are water compatible. The monomer structure includes hydroxyl groups, which imparts to these monomers significant water compatibility. On the other hand, alkyl groups in the molecular structure lower the water compatibility. Other water-compatible monomers like N-vinyl pyrrolidone or methacrylamide could be used as additives, but their costs are comparatively much higher. Small amounts of these materials, as additives, can significantly improve wet adhesion. No such study could be carried out within the short time allocated to this program. However, future studies in this direction could be carried out for further improvement in wet adhesion properties.

Ethyl hexyl acrylate and butyl acrylate were selected for evaluation because of their low cost and because their incorporation in minor amounts can moderate extremely glassy, brittle acrylics toward a slightly flexible, tougher material.

DOMA combined with tetraethylene glycol diacrylate (TTEGDA) was known to produce good flexural strength and other desirable properties suitable for rapid runway repair. However, in view of the suspected mutagenicity of TTEGDA,its replacement was necessary. TTEGDA is a cross-linking agent. Hexanediol diacrylate (HDDA), tetraethylene glycol dimethacrylate (TTEGDMA), pentaerythritol triacrylate (PETA) and tripropylene glycol diacrylate (TRPGDA) were selected for evaluation to replace TTEGDA. Acrylic prepolymers were also considered as cross-linkers and replacements for TTEGDA.

2. SMALL SPECIMEN COMPOSITIONAL INVESTIGATION

Castings of silica aggregates with selected monomers were carried out with percolation techniques using a number of compositional variations under three environmental conditions, i.e., dry, wet, and water-submerged round 1/4 x 1/2-inch aggregate. To obtain the wet aggregate condition, the aggregates were soaked in water overnight and shaken over a strainer prior to use. To obtain the water-submerged aggregate condition, the aggregates were kept in water-filled molds. The acrylic mix was poured from the top. The monomer system cured after displacing the water. All curings were carried out at 70°F. Tables 2 through 4 represent results of these

TABLE 2. CURING OF ACRYLIC-BASED SYSTEMS WITH DRY SILICA AGGREGATES AT 70°F

Monomer[(a)] System, percent	Cross-Linking[(a)] Agent, percent	Benzoyl[(b)] Peroxide, percent	Dimethyl-p-[(b)] Toluidine, percent	Cure Time,[(c)] minutes	Exotherm, °F	One Hour Flexural Strength, (d) psi
HEMA-HPMA[(e)] 20-60%:20-60%	HDDA/20	0.8	0.3	3-5	120-160	800-950
HEMA-HPMA 1:1	HDDA/20	0.2-0.8	0.3	3-10	100-150	1000-1300
HEMA-HPMA 1:1	TTEGDMA/5-20	0.6	0.3	2-3	130-140	800-1000
HEMA-HPMA 1:1	PETA/5-20	0.6	0.3	2-3	120-150	300-500
HEMA-HPMA 30-60%:20-50%	HDDA/20	0.6	0.3	4-6	145-165	1000-1800

(a) Relative proportions of monomers, including cross-linking agent, percent by volume.
(b) Weight percent initiator system based on monomers.
(c) To peak exotherm, which corresponds to setting time.
(d) Measured on 1 x 1 1/2 x 4 1/2-inch bars that were 30 volume percent resin, 70 volume percent of 1/4 x 1/2-inch round silica aggregate (generally average of three tests); at room temperature after 1 hour.
(e) Observed flexural strengths fall within indicated values and limit of precision.

HEMA - hydroxyethyl methacrylate
HPMA - hydroxypropyl methacrylate
HDDA - hexanediol diacrylate
PETA - pentaerythritol triacrylate
TTEGDMA - tetraethylene glycol dimethacrylate

TABLE 3. CURING OF ACRYLIC-BASED SYSTEMS WITH WET[(a)] SILICA AGGREGATES AT 70°F

Monomer[(b)] system, percent	Cross-Linking[(b)] Agent/Percent	Benzoyl[(c)] Peroxide, percent	Dimethyl-p-[(c)] Toluidine, percent	Cure Time,[(d)] minutes	Exotherm, °F	Flexural Strength,[(e)] psi
HEMA-HPMA 30-60%:20-50%	HDDA/20	0.6	0.3	5-7	130-150	700-950
HEMA-HPMA 20-60%:20-60%	HDDA/20	0.8	0.3	4-5	110-130	650-900

(a) Saturated surface wet (approximately 3 weight percent water).
(b) Relative proportions of monomers, including cross-linking agent, percent by volume.
(c) Weight percent initiator components based on monomers.
(d) To peak exotherm, which corresponds to setting time.
(e) Measured on 1 x 1 1/2 x 4 1/2-inch bars that were 30 volume percent resin, 70 volume percent of 1/4 x 1/2-inch round silica aggregate (generally average of three tests); at room temperature after 1 hour.

HEMA - hydroxyethyl methacrylate
HPMA - hydroxypropyl methacrylate
HDDA - hexanediol diacrylate

TABLE 4. CURING OF ACRYLIC-BASED SYSTEMS WITH WATER SUBMERGED SILICA AGGREGATES AT 70°F

Monomer[(a)] System, percent	Cross-Linking[(a)] Agent/Percent	Benzoyl Peroxide,[(b)] percent	Dimethyl-p-Toluidine,[(b)] percent	Cure Time,[(c)] minutes	Exotherm, °F	Flexural Strength,[(d)] psi	Remarks
HEMA	HDDA/15-20	0.6	0.3	17-19	55-70	230-270	
HPMA	HDDA/15-20	0.6	0.3	4-5	95-105	265-400	
HEMA-HPMA 80-84%, 3:1	HDDA/16-20	0.48-0.6	0.2-0.3	6-13	75-90	300-550	
HEMA-HPMA 84%, 3:1	HDDA/16	0.46-0.58	0.2-0.3	5-13	70-9	500-650	Triton X-100[(e)] [illegible] percent

(a) Relative proportions of monomers, including cross-linking agent, by volume.
(b) Weight percent initiator components, based on monomers.
(c) To peak exotherm, which corresponds to setting time.
(d) Measured on 1 x 1 1/2 x 4 1/2-inch bars that were 30 volume percent resin, 70 volume percent of 1/4 x 1/2-inch round silica aggregate (generally average of three tests); at room temperature after 1 hour.
(e) Added wetting agent to the monomer system.

HEMA - hydroxyethyl methacrylate
HPMA - hydroxypropyl methacrylate
HDDA - hexanediol diacrylate

experiments. It may be observed that the system HEMA-HPMA-HDDA, although it has a little higher exotherm, is quite comparable with similar DOMA systems shown in Table 5.

a. HDDA

The results reported in Tables 2 through 4 indicate that the use of hydroxyethyl methacrylate (HEMA), hydroxypropyl methacrylate (HPMA), and hexanediol diacrylate (HDDA) as an acrylic mixture results in fast-curing (2 to 10 minutes), strong aggregate composites. These systems obtained flexural strengths in the range of 1000 to 1300 psi* when composited with round dry silica aggregate (1/4 x 1/2 inch) and approximately 700 to 99 psi with wet silica aggregates. These values were within the same range of values as for DOMA-based acrylic systems. However, it was recognized that HDDA had a toxicity problem. HDDA has low reported values for acute toxicity, but is reported to be a severe skin sensitizer. As a result of this, the use of HDDA was abandoned. It was subsequently replaced by tripropylene glycol diacrylate (TRPGDA), which has very low reported values for all aspects of toxicity and flammability hazards. TRPGDA was an excellent acrylic cross-linker with other advantages shown in Table 1.

b. EHA and BA

Castings experiments were carried out with these monomers in combination with HEMA. In cases of wet and water-submerged castings they undesirably separated out from the mix after pouring because their specific gravity was less than one and they are hydrophobic. For this reason, they were not compatible with wet systems and hence they were not studied further.

c. TRPGDA

The performance of some systems based on HEMA, HPMA, and TRPGDA is shown in Tables 6 and 7. Fast cure times and reasonable exotherms were observed. All cures employed 0.5 to 1 percent benzoyl peroxide and 0.3 percent dimethyl-p-toluidine. That is, the exotherms were manageable and controllable, but sufficiently exothermic to provide acceleration to the cure. Flexural strengths were 850 to 1350 psi with the same dry silica aggregate. The use of certain additives such as acrylated polyesters

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* 1-inch by 1 1/2-inch by 4 1/4-inch test bars.

TABLE 5. CURING OF DOMA-BASED SYSTEMS WITH DRY SILICA AGGREGATES AT 70°F

	Cross-Linking(a) Agent/Percent	Benzoyl(b) Peroxide, percent	Dimethyl-p-(b) Toluidine, percent	Cure(c) Time, minutes	Exotherm, °F	Flexural Strength,(d) psi	Remarks
DOMA, 80-95	TTEGDMA/5-20	0.6	0.3	7-11	75-85	1000-1400	Effect of cross-linking agent and its concentration
DOMA, 80-85	PEGDMA/15-20	0.6	0.3	22-30	57-62	1400-1650	
DOMA, 80-95	PETA/5-20	0.6	0.3	4-10	75-100	400-550	
DOMA-HEMA, 80-95 2:1	PETA/5-20	0.6	0.3	3-5	100-125	300-700	
DOMA-HEMA, 80-95 2:1	TTEGDMA/5-20	0.5	0.3	4-5	115-130	850-1400	
DOMA-HEMA, 80-85 2:1	PEGDMA/15-20	0.6	0.3	11-17	80-90	2000-2100	
DOMA-HEMA, 80-85 2:1	HDDA/5-20	0.54	0.3	5-7	100	1200-1600	
DOMA-HEMA, 80 2:1	HDDA/20	0.2-0.8	0.3	5-12	80-130	950-1460	
DOMA-HEMA, 80 2:1	HDDA/20		0.1-0.4	4-11	93-115	1250-1650	
DOMA-HEMA, 80 5:3	HDDA/20	0.4-1.0	0.3	3-6	100-120	1500-2000	
DOMA-HEMA 10-60%:10-50%	HDDA/20		0.3	4.5-6	130-160	1000-1400	
DOMA-HEMA-HPMA, 80 2:3:3	HDDA/20	0.4-1.0	0.3	3-7	100-140	1500-1900	

DOMA - Dicyclopentenyl Oxyethyl Methacrylate
HEMA - Hydroxyethyl Methacrylate
HPMA - Hydroxypropyl Methacrylate
HDDA - Hexanediol Diacrylate

PETA - Pentaerythritol Triacrylate
TTEGDMA - Tetraethylene Glycol Dimethacrylate
PEGMA - Polyethylene Glycol Dimethacrylate

(a)-(d) Same as Table 2, page 8

TABLE 6. CURING OF ACRYLIC-BASED SYSTEMS WITH DRY SILICA AGGREGATES AT 70°F

Monomer System/(a) Percent	Cross-Linking(a) Agent/Percent	Benzoyl Peroxide,(b) percent	Dimethyl-p-Toluidine,(b) percent	Ebercryl, percent			Cure Time,(c) minutes	Exotherm, °F	Flexural Strength,(d) psi
				810	586	584			
HEMA/50	TRPGDA/10	1	0.3	30	--	--	5-6	135-145	1950-3200
HEMA/50	TRPGDA/10	0.8	0.3	40	--	--	6-7	115-140	2070-2450
HEMA/50	TRPGDA/10	0.8	0.3	--	40	--	3	155/170	120-240
HEMA/50	TRPGDA/10	0.8	0.3	--	--	40	3-4	155-175	1100-1300
HEMA-HPMA/1:1	TRPGDA/10	0.8	0.3	--	--	--	--	--	850-1350

(a) Relative proportions of monomers, including cross-linking agent, percent by volume.
(b) Weight percent initiator system based on monomers.
(c) To peak exotherm, which corresponds to setting time.
(d) Measured on 1 x 1 1/2 x 4 1/2-inch bars that were 30 volume percent resin, 70 volume percent of 1/4 x 1/2-inch round silica aggregate (generally average of three tests); at room temperature after 1 hour.

HEMA - hydroxyethyl methacrylate
HPMA - hydroxypropyl methacrylate
TRPGDA - tripropylene glycol diacrylate

TABLE 7. CURING OF ACRYLIC-BASED SYSTEMS WITH WET SILICA AGGREGATES AT 70°F

Monomer[(a)] System, percent	Cross-Linking[(a)] Agent/Percent	Benzoyl[(b)] Peroxide, percent	Dimethyl-p-[(b)] Toluidine, percent	Ebecryl[(a)] 810	Celanese[(a)] CMD 3201	Cure Time,[(c)] minutes	Exotherm, °F	Flexural Strength,[(d)] psi
HEMA-HPMA 1:1, 80%	HDDA/20	0.4-1	0.3	--	--	4-7	120-170	700-1150
HEMA-HPMA 1:1, 80%	HDDA/20	0.4-1	0.3	--	--	4-9	100-140	840-975
HEMA 60%	TRPGDA/10	1	0.3	30	--	4-5	150-160	1700-2200
HEMA 50%	TRPGDA/10	0.8	0.3	40	--	6-7	134-140	800-870
HEMA-HPMA 1:1, 80%	TRPGDA/20	0.8	0.3	--	--	--	--	870-1125
HEMA-HPMA 1:1, 80%	TRPGDA/20	0.8	0.3	--	20	--	--	800-1250
HEMA-HPMA 2.0:1.5, 90%	TRPGDA/10	0.8	0.3	--	20	--	--	850-1650
HEMA-HPMA 1:1, 80%	TRPGDA/20	0.8	0.3	20	--	--	--	700-1300

(a) Relative proportions of monomers, including cross-linking agent, percent by volume.
(b) Weight percent initiator system based on monomers.
(c) To peak exotherm, which corresponds to setting time.
(d) Measured on 1 x 1 1/2 x 4 1/2-inch bars that were 30 volume percent resin, 70 volume percent of 1/4 x 1/2-inch round silica aggregate (generally average of three tests); at room temperature after 1 hour.

HEMA - hydroxyethyl methacrylate
HPMA - hydroxypropyl methacrylate
HDDA - hexanediol diacrylate
PETA - pentaerythritol triacrylate
TTEGDMA- tetraethylene glycol dimethacrylate

(Ebercyl® 810) provided even higher strength. The cost of these materials is in excess of $2 per pound, and, in the amounts used, they were not cost effective.

d. Cost

Acrylic mixes of 20 percent TRPGDA and 80 percent HEMA-HPMA would result in a material cost of $1.16 to $1.20 per pound. This is based on price quotations of $1.35 to $1.50 per pound for TRPGDA and $1.13 per pound for HEMA and HPMA. Thus, substantial savings can be realized in potential material costs compared to DOMA-based acrylics.

e. DBP

To reduce costs and impart flexibility, a new additive, dibutyl phthalate (DBP) was used in the alternate acrylic castings. The performance of some systems based on HEMA-HPMA-TRPGDA and the new additive, dibutyl phthalate (DBP), is shown in Table 8. All cures were carried out at 70°F and employed 0.8 percent benzoyl peroxide and 0.3 percent dimethyl-p-toluidine. Dry 1/4-by 1/2-inch silica aggregate was used for all the cures. On curing, flexural strengths ranged from 1230 to 2672 psi which is significantly higher than previous systems without DBP. The use of 20 percent DBP produced the best flexural strength. Simultaneously, the materials cost of the system comes down to $1.01 per pound, based on the bulk price of DBP as $0.53 per pound. It can be seen that DBP offers a number of advantages, including high-cure strength, low hazard, and reduced cost.

f. Allyl Alcohol

One other approach to lower the cost of materials was the use of allyl alcohol in combination with the system described above. Allyl alcohol represents one of the least expensive liquid monomers available. Its incorporation during curing of the present system would help in improving strength and would effect considerable reduction in cost. However, its use with HEMA-HPMA-TRPGDA-DBP and HEMA-TRPGDA-DBP systems yielded very poor strength (Table 9). Apparently, the presence of allyl alcohol did not allow the polymerization of the system to proceed to completion.

g. Liquid Initiator

In a different set of experiments, systems consisting of HEMA-HPMA-TRPGDA-DBP were successfully polymerized with a new liquid initiator system

TABLE 8. EFFECT OF DIBUTYL PHTHALATE ON THE CURING OF ACRYLIC-BASED SYSTEMS

Percent[(a)] HEMA	Percent[(a)] HPMA	Percent[(a)] TRPGDA	Percent[(a)] DBP	Percent[(b)] BzP	Percent[(b)] DMPT	Flexural Strength[(c)], psi
40	40	10	10	0.8	0.3	1575
40	40	10	10	0.8	0.3	1442
35	35	10	20	0.8	0.3	2672
35	35	10	20	0.8	0.3	2646
30	30	10	30	0.8	0.3	1365
30	30	10	30	0.8	0.3	1230

(a) Percent by volume of total mix, excluding initiator.

(b) Weight percent based on monomer and DBP mix.

(c) Round dry silica aggregate with 70 volume percent voids, all cures were 3 to 7 minutes at 70°F; 1-inch by 1 1/2-inch by 4 1/4-inch beams; average of three tests; test at one hour.

TABLE 9. CURING OF ACRYLIC-BASED SYSTEMS WITH ALLYL ALCOHOL

Percent[(a)] HEMA	Percent[(a)] HPMA	Percent[(a)] TRPGDA	Percent[(a)] DBP	Percent[(a)] Allyl Alcohol	Percent[(b)] BzP	Percent[(b)] DMPT	Flexural Strength[(c)], psi
30	30	10	10	20	0.8	0.3	63
30	30	10	10	20	0.8	0.3	68
60	--	10	10	20	0.8	0.3	78
60	--	10	10	20	0.8	0.3	78

(a) Percent by volume of total mix, excluding initiator.

(b) Weight percent based on monomer and DBP mix.

(c) Round dry silica aggregate with 70 volume percent voids; all cures were 3 to 7 minutes at 70°F; 1-inch by 1 1/2-inch by 4 1/4-inch beams; test at one hour.

which we considered as a more suitable initiator system to replace pure granular benzoyl peroxide. This initiator system would be a pumpable liquid down to -30°F, would be safer, have an increased shelf life, and possess catalyst potential identical to benzoyl peroxide. This initiator system contains 27.5 percent dibutyl peroxide, 68.4 percent dibutyl phthalate, and 4.1 percent dibenzoyl peroxide. Both dry and wet 1/4-by 1/2-inch silica aggregate were used. Experiments were carried out at 70°F using 4 percent and 6 percent of this liquid initiator system. The accelerator used for this initiator was a 1:1 mixture of DMPT and cobalt napthenate. Results for both dry and wet silica aggregates are presented in Table 10. Flexural strengths for individual samples made with dry aggregates ranged from 1970 to 2691 psi and, for wet aggregates, the corresponding flexural strengths ranged between 360 and 1200 psi. These systems contained 10 percent DBP but (as shown in Table 8) the flexural strength might be improved further with the use of 20 percent DBP. In view of the short duration of this program, detailed work with the liquid initiator system could not be carried out to effectively use it in larger castings. Work on this aspect can be extended in the future.

h. Curing Without Peroxide Initiator

From an in-house study at Battelle, we have developed a new curing system in which no conventional initiators like peroxides, AZO compounds etc. are required. Standard chemicals in combination with suitable monomers can initiate the polymerization. Preliminary experiments with silica aggregates and HEMA system could cure within 5-7 minutes at 70°F and flexural strength at 1 hour was about 1000 psi for large beams (4 inches by 4 inches by 14 inches). Work on this may also be pursued in the future.

3. LARGE SPECIMEN TESTS

Screening experiments with castings of small beams 1 inch by 1 1/2 inches by 4 1/4 inches containing 1/4-by 1/2-inch quartz aggregate and (HEMA-HPMA-TRPGDA) systems, obtained good flexural strengths and fast cure rates, comparable to DOMA systems. However, as has been observed earlier, the strength obtained for large beams measuring 4 inches by 4 inches by

TABLE 10. CURING OF ACRYLIC-BASED SYSTEM USING LIQUID INITIATOR SYSTEM

Type of Aggregate	Percent(a,c) Liquid Initiator	Percent(b) HEMA	Percent(b) HPMA	Percent(b) TRPGDA	Percent(b) DBP	Percent(c) DMPT	Percent(c) Cobalt Napthenate	Flexural(d) Strength, psi
Dry 1/4 x 1/2 inch silica	4	35	35	20	10	0.4	0.4	2200
	4	35	35	20	10	0.4	0.4	2350
Wet 1/4 x 1/2 inch silica	4	35	35	20	10	0.4	0.4	937*

(a) Liquid Initiator - 27.5 percent dibutyl peroxide
68.4 percent dibutyl phthalate
4.1 percent dibenzoyl peroxide.

(b) Relative percent, by volume, of monomer - DBP mix.

(c) Percent (by volume) Initiator, Percent DMPT, Percent CoNAP are based on monomer - DBP mix.

(d) Average of three tests conducted on 1 x 1 1/4 x 4 1/4-inch bars; 70°F test at 1 hour.

* (360-1200 psi range)

14 inches produced 1-hour strength values about half those obtained with small beam tests. Several factors including (1) the dissipation of heat (related to the sample surface area-to-volume ratio), (2) aggregate and specimen size, and (3) test method might be responsible for such differences. The major factors are considered to be the differences in heat dissipation and test method. Whereas small beams substantially cooled within 1 hour, large beams remained at significantly high temperatures after 1 hour. The fracture characteristics for large beams indicated that the beams under stress break in a yielding pattern. This soft character of the large beams was attributed to the fact that the temperature of the beam exceeded the glass transition temperature (Tg) at the time of testing. The test method could also be a factor because the small bars were broken in three-point flexure (rough top surface in compression) while the large beams were broken in four-point flexure (ASTM C78).

Table 11 summarizes a typical set of experiments carried out with No. 57(a) quartz aggregate and cast beams 4 inches by 4 inches by 14 inches in dimension. The results presented are average of three values for each type of experiment. To obtain the wet aggregate condition, the aggregates were soaked in water overnight and shaken over a strainer prior to use. Results shown in Table 11 indicate that HEMA,in combination with TRPGDA, might produce a higher flexural strength for the wet aggregate condition after 1 hour than that obtained from HEMA-HPMA-TRPGDA system in combination with 10 percent DBP. The fracture characteristics indicated that the beams under stress break in a yielding pattern. Hence, to decrease the soft character, TRPGDA was reduced further and DBP completely eliminated to increase the Tg of the final polymer. The results for such systems are presented in Table 12. As expected, reduction of TRPGDA concentration significantly increased the 1-hour flexural strength, when wet aggregate was used.

Further testing of similar compositions was performed at 125°F and -20°F with large beam castings. The BzP level was adjusted for these temperatures based on prior experience. For wet aggregates at 125°F the aggregates were soaked in water at 125°F overnight. For wet aggregates at -20°F the

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(a) ASTM C33 (nominally 1-inch by 4-mesh material).

TABLE 11. FLEXURAL STRENGTH OF ACRYLIC-BASED SYSTEMS AT 70°F USING NO. 57 QUARTZ AGGREGATE

Aggregate Condition	Percent(a) HEMA	Percent(a) HPMA	Percent(a) TRPGDA	Percent(a) DBP	Percent(b) BzP	Percent(b) DMPT	Flexural Strength,(c) psi at 1 hour
Dry	90	-	10	-	0.6	0.2	985
Wet	90	-	10	-	0.6	0.2	360
Wet	35	45	10	10	0.6	0.2	241

(a) Relative proportions, by volume, of monomer - DBP mix.
(b) Weight percent initiator components, based on monomer - DBP mix.
(c) Casting size 4 inches by 4 inches by 14 inches; average of three experiments for each type.

TABLE 12. FLEXURAL STRENGTH OF ACRYLIC SYSTEM AT LOW CONCENTRATIONS OF TRPGDA AT 70°F USING NO. 57 QUARTZ AGGREGATE

Condition of Aggregate	Percent(a) HEMA	Percent(a) HPMA	Percent(a) TRPGDA	Percent(b) BzP	Percent(b) DMPT	Flexural Strength,(c) psi at 1 hour
Dry	95.5	-	4.5	0.6	0.2	850
Wet	95.5	-	4.5	0.6	0.2	510
Wet (d)	95.5	-	4.5	0.6	0.2	585
Dry	97.7	-	2.3	0.6	0.2	920
Wet	97.7	-	2.3	0.6	0.2	510
Wet	47.8	47.7	4.5	0.6	0.2	542

(a) Relative proportions, by volume, of monomer.
(b) Weight percent initiator components, based on monomer.
(c) Casting size 4 inches by 4 inches by 14 inches; average of three experiments for each type.
(d) Aggregate treated with silane coupling agent.

aggregates were stored overnight at 5°F and poured in ice-cold water prior to curing. In both the cases, the aggregates were shaken over a strainer just before curing. The results are shown in Tables 13 and 14.

Tables 13 and 14 show that the improved acrylic system based on HEMA-TRPGDA is equally good at -20°F and 125°F. This system can provide the desired strength at all test temperatures for both dry and wet aggregate conditions. Furthermore, no additional treatment of aggregate, such as coupling agents or any additive, was required to obtain the indicated strengths for either dry or wet aggregates. The viscosity of the monomer system is approximately 7 cps at 70°F and the material can easily percolate into aggregate under all environmental conditions. Experiments were carried out under conditions of aggregate beds submerged in water, wet aggregates under simulated rain, and surface-saturated wet aggregates. Under all the above mentioned conditions, the system exhibited the capability of displacing water from the aggregate body and curing to a high-strength material.

Work on the HEMA-TRPGDA system was extended under the ongoing Rapid Runway Repair program of AFESC. Brief mention of some of this work is made here to indicate the potential of this system. All castings were 4 inches by 4 inches by 14 inches in dimension. This work includes:

a. Wet No. 57 quartz aggregate at 70°F placed in a half inch of water at the bottom of mold and under continuous rain simulation gave a 1-hour flexural strength of 656 psi.

b. Premixing of dry No. 57 quartz aggregate, 30-by-100 mesh glass sand and HEMA-TRPGDA system at 70°F (cure time was varied between 20 to 30 minutes for handling convenience) gave a 1-hour flexural strength of 1700 psi.

c. Dry sand was percolated simultaneously with the HEMA-TRPGDA system into both dry and wet aggregate to obtain flexural strengths as follows: 70°F, dry, 1150 psi; 70°F, wet, 1300 psi; -20°F, wet, 700 psi.

TABLE 13. FLEXURAL STRENGTH OF ACRYLIC SYSTEM USING NO. 57 QUARTZ AGGREGATE AT 125°F

(a) Percent HEMA	(a) Percent HPMA	(a) Percent TRPGDA	(b) Percent BzP	(b) Percent DMPT	Aggregate Condition	Flexural Strength,(c) psi at 1 hour
95	--	5	0.6	0.17	Wet	423
95	--	5	0.4	0.17	Dry	984
97.4	--	2.6	0.4	0.17	Wet	563
97.4	--	2.6	0.4	0.17	Wet	479
97.4	--	2.6	0.6	0.17	Dry	995
49.5	49.5	5	0.4	0.17	Wet	271
49.5	49.5	5	0.4	0.17	Dry	725

(a) Relative proportions, by volume, of monomer.
(b) Weight percent initiator components, based on monomer.
(c) Casting size 4 inches by 4 inches by 14 inches.

TABLE 14. FLEXURAL STRENGTH OF HEMA-TRPGDA SYSTEM USING NO. 57 QUARTZ AGGREGATE AT -20°F

(b) Percent HEMA	(b) Percent TRPGDA	(c) Percent BzP	(c) Percent DMPT	Aggregate Condition	Flexural Strength,(a) psi at 1 hour
97.4	2.6	2	0.17	Wet	748
97.4	2.6	3	0.17	Wet	811
95	5	3	0.17	Wet	731
95	5	2	0.17	Wet	597
95	5	3	0.17	Dry	750
95	5	3	0.17	Wet/Silane	849

(a) Relative proportions, by volume, of monomer.
(b) Weight percent initiator components, based on monomer.
(c) Casting size 4 inches by 4 inches by 14 inches.

SECTION III
ASSESSMENT

1. CONCLUSIONS

The goal of this short research program was to achieve a basic improvement in the water compatibility of acrylic materials and to utilize lower cost acrylic materials, while maintaining the desirable characteristics achieved with the DOMA system: rapid strength development over the full environmental range, high strength and toughness, low viscosity, minimum flammability and toxicity hazards, long shelf life, and ease of handling.

The major objectives of improving water compatibility and reducing costs were achieved with the identification of hydroxyethyl methacrylate (HEMA) resin. The introduction of HEMA achieved approximately 100 percent better wet strength and reduced resin costs to approximately \$1.13 per pound,as compared to \$2.17 per pound for DOMA. The HEMA-TRPGDA system obtained good strength over the entire range of environmental conditions set forth for the program. However, this was a limited study of short duration. The results can be considered a preliminary identification and development of an alternative acrylic curing system that offers substantial advantages over the DOMA system. The system could be improved considerably with further studies to incorporate small amounts of dibutyl phthalate and N-vinyl pyrrolidone. The former provides a tougher, more craze-resistant material, while the latter obtains improved wet adhesion. A balance of the two is required for good wet adhesion to rock while preventing crazing at low temperatures (not observed during this program, but seen during other related work) and maintaining high modulus at higher temperatures. Further work on this is recommended.

2. RECOMMENDATIONS

The HEMA-TRPGDA system formulation range suggested by the results of the current program is:

a. HEMA - 95 to 97.5 percent

b. TRPGDA - 2.5 to 5.0 percent

c. Benzoyl Peroxide (Paste) - 0.3 to 3 weight percent active peroxide content based on the total HEMA-TRPGDA-DBP volume

d. DMPT - 0.2 weight percent of the total HEMA-TRPGDA-DBP volume.

As mentioned above, a small amount (e.g., 2.5 percent) of DBP additive should also be considered, based on results in other related work.

Introduction of minor amounts of dibutyl phthalate may improve the flexural strength and reduce crazing, but lower wet adhesion unless small amounts of N-vinyl pyrrolidone are also added to the monomer mix. Other improvements may be obtained in future research with the use of the previously mentioned liquid initiator system. This could provide a pumpable initiator system. Another promising approach would be to develop a cure system without peroxides or AZO initiators that is capable of rapidly curing to high strength. This would reduce the hazards involved with the use and storage of dibenzoyl peroxide.

Preliminary experiments reported earlier on other modes of applications of HEMA-TRPGDA system, i.e., premixing of aggregate and sand and percolation of sand with the monomer mix,show promise of achieving even higher strength than with the coarse aggregate alone. The inclusion of sand in the casting more than doubles the cure strength. Simultaneously, it reduces the void volume significantly and gives lower material costs.

It is therefore recommended that future studies with this system be carried out in the following directions:

a. Curing of HEMA-TRPGDA system in the absence of conventional initiators such as peroxides or AZO compounds. This is possible by combination of standard chemicals (see p. 18) which are completely inert by themselves but allow rapid polymerization when they are combined. There are a number of advantages in such initiator-less cure systems. The hazards of handling and storage are eliminated. No special storage conditions or facilities are needed as is the case with peroxides, thereby reducing costs for storage facilities.

b. Studies on the optimization of dibutyl phthalate. Dibutyl phthalate acts as a plasticizer in HEMA-TRPGDA system. It increases flexural strength, decreases modulus and is capable of reducing crazing during rapid curing, especially at low temperatures. It can reduce resin-to-aggregate wet adhesion. Further studies are needed to balance the opposing effects of wet adhesion and plasticization. This would be particularly valuable optimization and refinement for application over all environmental conditions.

c. Premixing of aggregate, sand, and monomer system. The HEMA-TRPGDA system, with a suitable initiator, can be premixed with aggregate and sand. To allow for sufficient mixing and handling of such a premixed system, cure times should be adjusted to approximately 15 to 30 minutes. Preliminary experiments on the premix approach produced flexural strengths over 1700 psi. The void volume dropped to approximately 15 percent, thereby reducing by half the monomer mix needed for percolation. This technique effectively reduces cost of materials and yields higher strength polymer concrete. It must also be noted that preliminary premix tests using this system with wet aggregate yielded flexural strengths of negligible value.

d. Percolation of sand. In another approach dry sand was percolated with the monomer mix into the quartz aggregate. This approach also reduced the quantity of cure mix needed and increased flexural strength of the cure material. The approach was tested both under dry and wet aggregate conditions. In both cases, the strength obtained after 1 hour ($\sim$1300 psi) was significantly higher than with the usual percolation of cure mix alone into aggregate. This could also aid in cost reduction and increase strength properties.

3. SAFETY

One of the major objectives in this program was to select materials having the least hazards for personnel performing the bomb damage repair. Tables 15 and 16 list some of the basic properties of materials used. It may be observed that HEMA, TRPGDA, DBP, and BzP are low in toxicity. Except for BzP, all components are low volatility, low flammability liquids. DMPT has moderate toxicity but is used in very small quantity (0.2 percent).

TABLE 15. PROPERTIES OF MONOMERS

	DOMA	HEMA	TRPGDA	MMA(a)
Boiling Point, °F	662	386	>514	214
Melting Point, °F	--	<-40	<32	-54
Vapor Pressure, MM Hg	<0.1 (77°F)	0.01 (77°F)	0.001 (68°F)	40 (77°F)
Viscosity, cps	15-19 (77°F)	5.5 (86°F)	11.9 (77°F)	--
Density, g/cc	1.064	1.070	1.03	0.94
Flash Point, °F	>200	214	>200	49
pH	Neutral	Neutral	Neutral	Neutral
Acute Oral LD_{50}, Mg/Kg	>5000	5000	6800	7900
Eye Irritation	Nonirritating	Mild(b)	Slight(b)	--
Skin Irritation	Draize 1-2	--	Mild(c)	--
Acute Inhalation	Nontoxic	Mild(b)	Mild(b)	12,500 ppm
Mutagenicity	Nonmutagenic	--	--	--
Skin Sensitizer	Nonsensitizing	--	--	--
TLV, ppm	--	--	--	50

(a) By way of comparison to a common volatile monomer.

(b) Mild because of low vapor concentration.

(c) Mild after 72 hours of contact.

TABLE 16. SAFETY DATA AND COSTS OF MONOMERS AND ADDITIVES

Monomer	Flash Point, °F	Toxicity	Flammability	Price/lb
DOMA	200	Low	Low	$2.07
HEMA	214	Low	Low	1.13
TRPGDA	200	Low	Low	1.35
HPMA	208	Low	Low	1.15
MMA	47	High	High	0.60
HDDA	200	(Skin Sensitizer)	Low	1.50
PEGDMA	200	Low	Low	2.14
Other				
Benzoyl Peroxide (Initiator)	221 (Store ≤ 85)	Low	(Oxidizer)	1.88
DMPT (Accelerator)	208	Moderate	Low	
Dibutyl Phthalate (Plasticizer)	340	Low	Low	0.56

Although these materials are low in toxicity, it is recommended that users wear protective clothing, including gloves and goggles. This ensures protection against direct splash contact. High vapor concentrations are not possible with these chemicals at ambient conditions and respirators are not required.

In the event of accident involving large quantities of material on the skin, clothing, or eyes, the affected parts should be thoroughly washed with plenty of water followed by a physician's examination. In case of accidental ingestion, vomiting should be induced and medical attention obtained.

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