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DEVELOPMENT OF A PROTOTYPE COMPOSITE FIELD REPAIR KIT

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SOUTHERN RESEARCH INSTITUTE

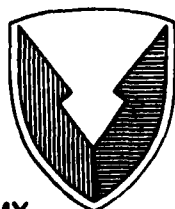
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ABSTRACT

A novel composite field repair kit concept based on packaging utilized in the instant photography field has been investigated. The objective of the work was to retain the simplicity of a one-part patch while circumventing the shelf-life problems associated with such materials. In essence, the kit would isolate each component until the patch was needed and then combine them in the proper ratio by means of a simple operation. A number of kit designs were investigated along with several variants of the basic materials formulation. While an acceptable kit was developed, its components could not be adequately mixed without extensive manipulation due, in part, to a severe mismatch in their viscosities. Thus, the kit did not constitute a significant advance over similar state-of-the-art kits.

CONTENTS

	Page
1. INTRODUCTION.	1
2. MATERIALS AND METHODS	1
2.1 Selection of Packaging Materials	2
2.2 Resin/Hardener Compositions.	2
2.3 Kit Design	3
2.4 Fabrication of Prototypes.	5
2.5 Evaluation of Prototypes	6
3. RESULTS AND DISCUSSION.	8
3.1 Study of Mixing-Chamber Design	8
3.2 Effect of Liquid-Components-Reservoir Design	11
3.3 Effect of Resin and Hardener Composition on Mixing	11
3.4 Effect of Processor Speed.	11
3.5 Effect of Multiple Passes on Mixing.	13
3.6 Fibrous Pad Containing Hardener.	13
3.7 Effect of Mixing With a Hand Roller.	14
3.8 Pot-Life Studies	15
4. CONCLUSIONS AND RECOMMENDATIONS	16
5. ACKNOWLEDGMENT.	16



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1. INTRODUCTION

The objective of this project was to design and develop a composite field repair kit containing the three required components: epoxy resin, amine curing agent, and glass cloth. The need for making repairs easily and quickly in the field precludes weighing the required amounts of epoxy resin and hardener, mixing the two components, and subsequently impregnating a reinforcing patch with the mixed components. Rather, a kit is preferred in which the individual components are premeasured and stored separately so that the kit will have a long shelf life, be easy to use, and be activated by a simple processor. The kit would then be passed through the processor, and upon opening the package, the impregnated patch would be removed and placed upon the composite structure to be repaired. Ideally, the processor should be similar to those used in the "instant-photography" field.

Toward this end, Southern Research Institute undertook a research project to design and develop a kit and processor that would meet these requirements. The program was divided into three phases. Phase I included the development of concept models, selection of packaging materials, and design validation. The design validation included, as a minimum, the following four parameters.

- Thoroughness of mixing
- Uniformity of spreading and impregnation of the patch
- Effectiveness and simplicity of the processor
- Durability of packaging material

Upon completion of Phase I of the project, a review meeting was to be held to consider approval of the kit design. If the design was acceptable to the government, then Phase II was to be initiated.

In Phase II, prototype kits were to be fabricated and tested under the guidelines established in Phase I. At the end of Phase II, the prototype that was most satisfactory was to be demonstrated at a second review meeting.

Finally, after approval of the selected prototype, Phase III was to be initiated. In Phase III, we planned to fabricate 50 kits and two processors and deliver these to the government. Also, an instruction manual describing the use of the kit and processor was to be delivered to the government.

2. MATERIALS AND METHODS

Phase I of this project was directed toward both the selection of packaging materials and the design, fabrication, and evaluation of prototype repair kits. During this time, we investigated the durability of the packaging material, the pot life and curing time of various resin/hardener compositions, and the fabrication of a burstable seal. Also, we have studied the effects of the design of the liquid-component reservoirs and the mixing chamber on the thoroughness of mixing. This work is described in the following sections.

2.1 Selection of Packaging Materials.

In our evaluation of packaging materials, we examined their burst strength, temperature stability, ability to retain a vacuum, and inertness to the epoxy components. Because of the reactivity of the amine hardeners, polyolefin sheets and laminates with an inner lining of polyolefin were the only materials we evaluated. Our initial compatibility tests showed that polyethylene (PE) film was not affected after prolonged exposure to either the resin or amine hardeners. Because of the requirement for a long shelf life, we felt that PE alone did not offer adequate barrier properties against water vapor. For this reason, we considered only laminates consisting of an inner polyolefin layer bonded to a layer of aluminum foil. Two materials were chosen for evaluation. One was a polyethylene/aluminum foil/kraft paper laminate, AT5031, from Ludlow Corporation, Holyoke, MA. The other was a polypropylene/aluminum foil/nylon laminate, Marvelseal 360, also from Ludlow Corporation.

Twenty samples of each of these materials were tested on a Mullen Burst Tester. The average burst strength for the AT5031 laminate was 47 psi while that of the Marvelseal 360 was 78 psi. Although the Marvelseal 360 appeared to be the better choice, we found that it would require thermo-stripping for permanent heat sealing. Consequently, we used the AT5031 in all subsequent work.

2.2 Resin/Hardener Compositions.

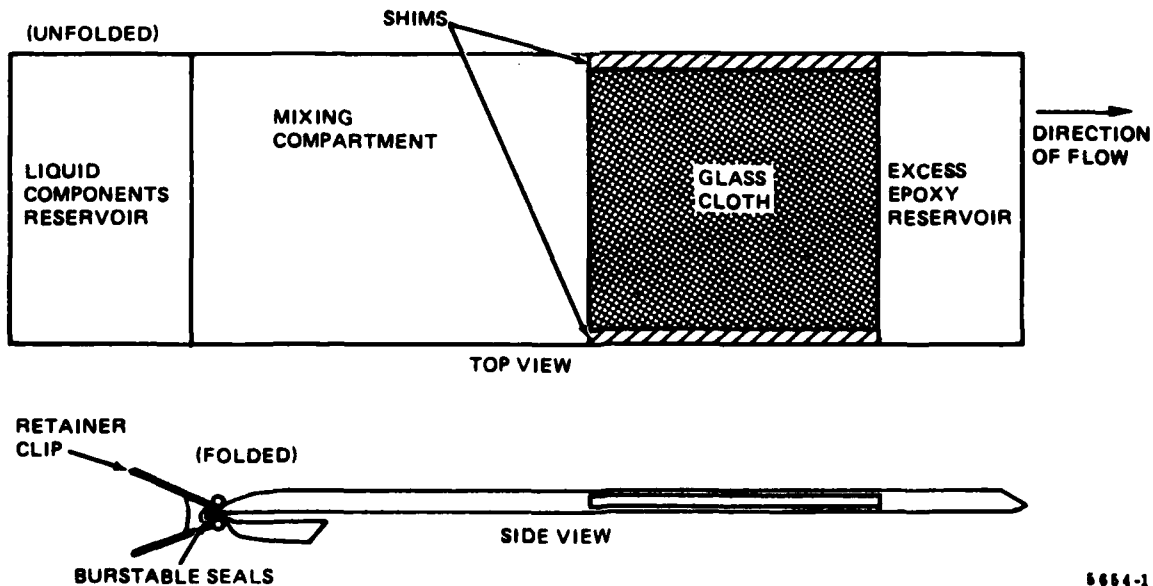
All of the work performed on the project was centered on the use of Epon 828 epoxy resin and diethylenetriamine (DETA) both of which were obtained from Shell Chemical Company, Houston, TX, these materials were those those specified in the contract. During our preliminary studies, however, we decided that better mixing could be achieved by modifying the resin/hardener system. This decision was based on two considerations. First, the Epon 828 resin is considerably more viscous than the DETA, and the disparity between the viscosities increases the difficulty of mixing the two materials. Furthermore, the resin/hardener volume ratio for the Epon 828/DETA system is about 10 to 1, which does not favor good mixing. To reduce these effects, we evaluated two other materials in our work. The first material that we considered was Araldite RD-2 Reactive Diluent (butyl glycidyl ether, Ciba Geigy, Ardsley, NY). Araldite RD-2 is commonly used to reduce the viscosity of epoxy resins and is reported to have a minimal effect on the strength of the cured resin. The second material that we used was Curing Agent U (Shell Chemical Company, Houston, TX). Curing Agent U is an amine adduct that is essentially the product formed by capping Epon 828 with DETA. Because of the longer chain length of this material, it has a much higher viscosity than DETA. Moreover, Curing Agent U has a higher amine equivalent weight than DETA, and the resin/hardener volume ratio is lower for Curing Agent U than for DETA. The resin/hardener systems that we investigated are listed below.

- Epon 828/DETA
- Epon 828/Curing Agent U
- Epon 828:Araldite RD-2/DETA
- Epon 828:Araldite RD-2/DETA:Curing Agent U

The results obtained with these systems are discussed in Section 3 of this report.

2.3 Kit Design.

Our original concept for the repair kit included a resin compartment, hardener compartment, mixing chamber, and a fabric compartment. The resin and hardener compartments were to be sealed with "burstable" seals, as was the fabric compartment. The pressure generated upon passing the kit between the two rolls of a processor would burst the seals of the resin and hardener compartments and force these materials into the mixing chamber. During transit in the mixing chamber, the materials would be mixed. At the exit end of the mixing chamber, a second seal would be ruptured, and the mixed resin would be forced onto the glass cloth. We conducted our initial design studies with a clear packaging material so that we could observe the mixing of the resin and hardener during processing. In those tests, we doped either the resin or hardener with a colored pigment. These studies showed that mixing was not easily achieved because of the relatively high viscosity of the epoxy resin. Also, we determined that the kit should consist of four distinct sections. The four sections are a liquid-components reservoir, a mixing chamber, a patch compartment, and an excess-liquid reservoir. The general design of the kit is shown in Figure 1. Note the folded seal and clip arrangement between the liquid-components reservoir and the mixing chamber. This was determined to be the most reliable seal.



5654-1

Figure 1. Schematic of prototype repair kit.

One approach to improved mixing that we considered was the arrangement of the resin and hardener compartments. Figure 2 shows the various designs that we evaluated. These range from simple two-compartment designs to a five-compartment design. While increasing the number of compartments improves the overall mixing, this alone does not solve all of the problems associated with mixing the two components.

Figure 3 shows the basic designs considered for the mixing chamber. The simplest of these is an open compartment with no baffles. A kit of this type was demonstrated to Dr. Wentworth during his visit on December 17, 1984. The remaining designs differ by the degree of complexity of the system of baffles included in the mixing chamber. The addition of baffles was done to increase the shear forces acting on the liquids during transit in the mixing chamber.

The glass-cloth compartment is relatively simple. Two shims are included to control the amount of resin that is impregnated into the cloth. These are constructed to hold the fabric in place and prevent movement of the fabric during processing. The excess-resin reservoir was included to contain excess resin and prevent rupture of the kit at the downstream end. Excess resin is required to obtain uniform impregnation of the fabric.

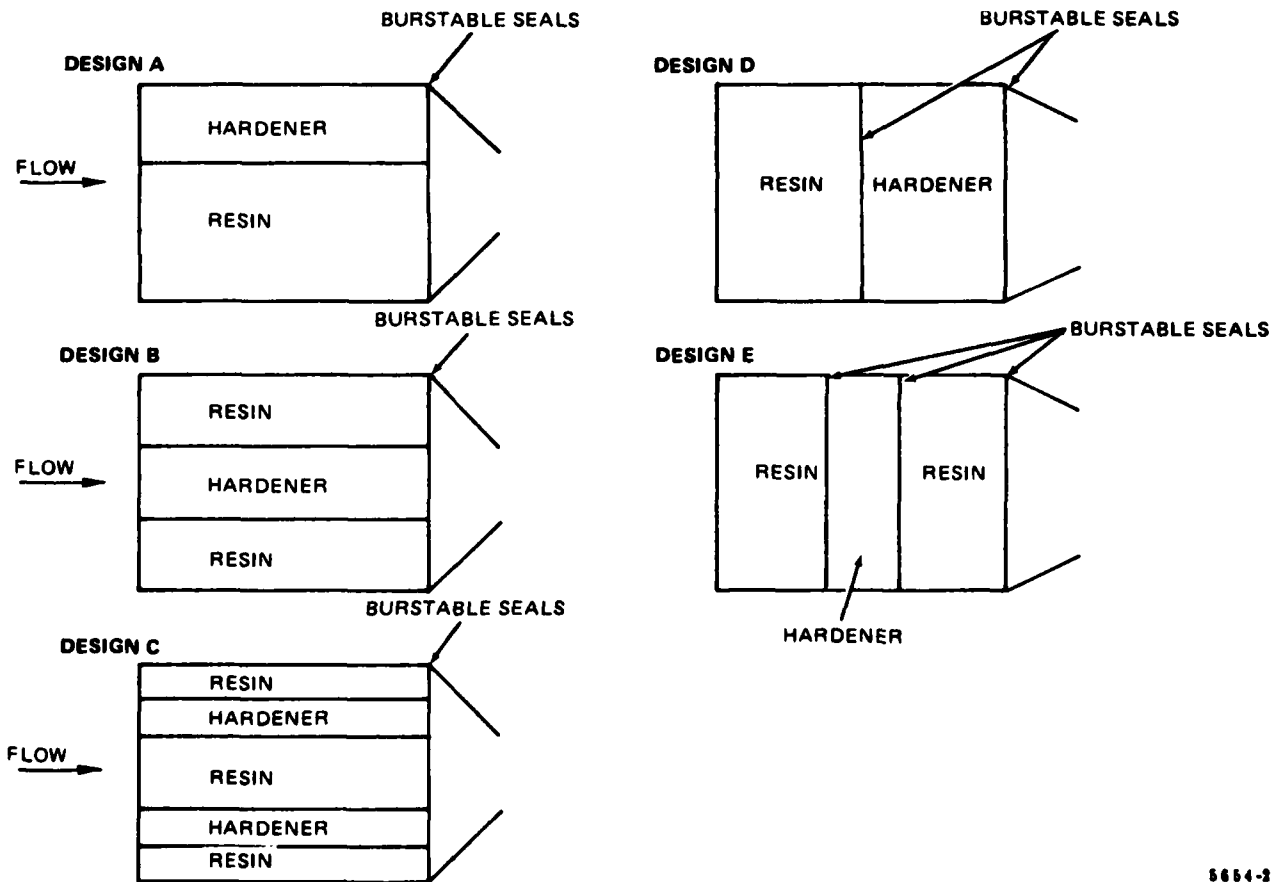
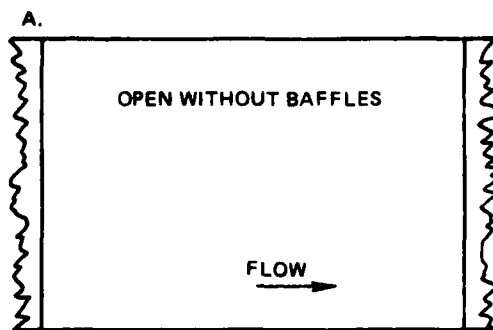
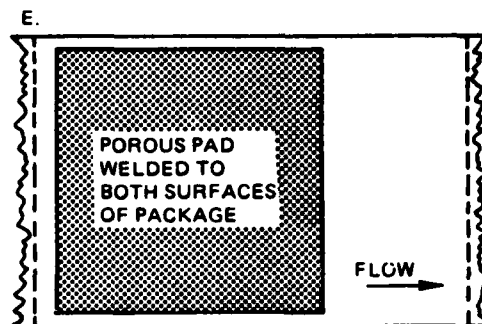
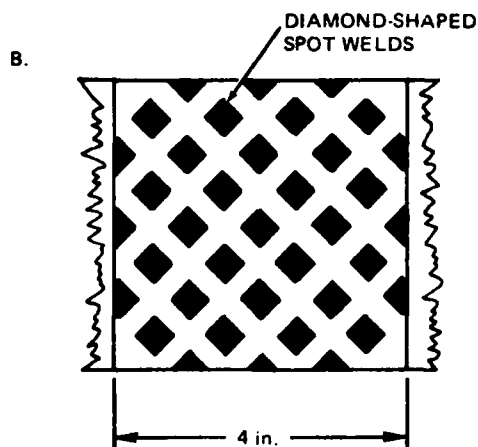


Figure 2. Designs for the epoxy-components compartment.



C. SAME AS B EXCEPT 4 x 8 in.

D. SAME AS B EXCEPT 4 x 12 in.



5654-3

Figure 3. Mixing-chamber designs.

2.4 Fabrication of Prototypes.

Except for our preliminary work, all prototype kits were manufactured using the AT5031 laminate from Ludlow Corporation. With the exception of the mixing-chamber pattern, all of the heat seals were made with a 1/8-in. ribbon-type thermal-impulse heat sealer from Vertrod Corporation. The mixing-chamber pattern shown in Figure 3.A was fabricated using a 4 x 4 x 1/4-in. aluminum template. Pattern A was produced by placing the two layers of AT5031 laminate over a 1/8-in. silicone-rubber pad resting on the bottom platen (unheated) of a Pasadena Heat-press (Pasadena Hydraulics Inc., El Monte, CA). The aluminum template was placed pattern down on top of the upper layer of material, and the press was closed so the 4 x 4-in. aluminum template contacted the top, heated platen of the press. The time, temperature, and pressure conditions required for the formation of a good bond between the two layers were determined.

The burstable seals were made with the Vertrod Heat sealer at a setting of 1.0 to 1.5 on the dwell dial. We attempted to prepare seals that were leakproof yet weak enough to rupture under the applied pressure of the processor. Unfortunately, seals that are strong enough to prevent leaking of

the hardener do not rupture reliably. We attempted to overcome this problem by using a strip of low-density PE such as Parafilm (Greenwich, CT) in the seal. This approach did not work well either. The best approach appears to be the use of a weak heat seal and a mechanical retainer clip to keep the less-viscous liquid components from leaking. The clip that best suited our requirements was a split length of rigid polyethylene tubing that we manufactured ourselves.

All seals except the end seal for the liquid-components reservoir were made before filling the resin and hardener compartments. The resin and hardener were added through the end of the liquid compartments, and the air was pressed out. The end of the kit was then heat sealed with the Vertrod heat sealer. All kits were allowed to sit overnight at ambient temperature before processing.

2.5 Evaluation of Prototypes.

2.5.1 Thoroughness of mixing.

One of the major tasks that was to be completed in Phase I was the development of a quantitative measure of the thoroughness of mixing. We examined four methods for this aspect of the project. Initially, we attempted to determine the degree of mixing by measuring the exothermic heat of reaction on a Perkin-Elmer differential scanning calorimeter. This method was extremely time-consuming, and the observed heat of reaction was highly dependent on the elapsed time between processing the kit and starting the scan. For these reasons, it was abandoned. The second method involved the use of two different dispersed pigments, one in each of the epoxy components. While this method provided a good qualitative assessment of the degree of mixing, it was only useful for rangefinding studies. In the third method, we dissolved an acid-base indicator in the resin which changed color when mixed with the hardener. This technique provides excellent sensitivity and is a good way to determine the degree of mixing both qualitatively and quantitatively. Unfortunately, a specific procedure is not generally applicable to a wide range of curing agents because the dye must be selected on the basis of the change of pH that occurs on mixing. For this reason, the technique requires considerable development time when a new resin or hardener system is used.

For the last method, we used a dye dissolved in the hardener. We chose Solvent Red 119 (SR 119) as the dye for these studies. Solvent Red 119 is another name for Neozapon Fire Red G from BASF Corp., Holland, Michigan. To quantify the amount of DETA present in resin/hardener mixtures, a Beer's Law plot of absorbance versus dye concentration was prepared by the following procedure.

We prepared a stock solution by dissolving 0.88 g of SR 119 in 202.9 g of DETA. This solution was used in the development of the assay procedure as well as in the preparation of prototype kits. We prepared a second stock solution by diluting 1.11 g of the SR 119/DETA solution to 250 mL with *n*-propanol. We prepared a third stock solution by dissolving 10.06 g of Epon 828 in enough *n*-propanol to yield 250 mL. Solutions comprising various ratios of the latter two stock solutions were prepared, and the absorbance was determined as a function of wavelength on a Perkin-Elmer Lambda 3b Scanning UV-VIS spectrophotometer. The maximum absorbance was observed at 454 nm.

Because the ratio of SR 119 to DETA is constant, the absorbance can be related to the DETA concentration in the n-propanol solution. A Beer's Law plot of absorbance at 454 nm versus the concentration of DETA in g/mL gave a correlation coefficient of 0.994, a Y-intercept of 0.00001, and a slope of 0.00902.

We were concerned about the potential effect on the absorbance of the cured epoxy. To determine this effect, we allowed the solutions containing SR 119, DETA, and Epon 828 to stand for 30 min. The scans were repeated, and no change in absorbance was observed. Upon further standing, however, a slight turbidity was noted. When the scans were repeated, a high absorbance was observed across the entire spectrum. From this information, we decided that the samples of processed kits should be read on the UV-VIS spectrophotometer within 30 min of processing. We then repeated the above procedure using the same two 250-mL stock solutions (which showed no signs of turbidity) and obtained identical results. This entire procedure was repeated later using an initial starting mixture of 1.02 g of Solvent Red 119 in 249.49 g of Curing Agent U to prepare a standard curve for use with kits prepared with the Curing Agent U hardener.

2.5.2 Test procedure.

In these experiments an Atlas laboratory wringer, type LW-1 (Atlas Electric Devices Co., Chicago, IL), was used to process the kits. A variable-speed DC motor was attached to the wringer, and the fixed-speed motor supplied with the wringer was detached. Most of the experiments were performed at 8 rpm (1 rpm = 0.55 linear ft/min), and all kits were processed at a 1295-g load on the 1-ft lever attached to the top roller of the wringer. The mechanical seals on the kit were removed (if present), and the kit was passed between the rollers, liquid compartment first. In cases where multiple passes were performed, the wringer was stopped 3 in. before the patch compartment, and the kit was turned around and passed back through the wringer. This procedure was repeated until the desired number of passes had been achieved.

Immediately after processing, the kit was placed flat on a Teflon sheet and 1-1/8-in. samples were stamped from the patch compartment using a 1-1/8-in.-diameter arch punch. Typically, five samples were removed from each kit. Each sample was placed in a preweighed scintillation vial containing 10 mL of n-propanol, and the vial was reweighed to determine the sample weight. The contents of each vial were mixed vigorously on a vortex mixer (Vortex-Genie Model No. K-550-G, Scientific Industries, Inc., Bohemia, NY). Finally, the absorbance at 454 nm was determined.

The weight of the resin/hardener mixture in each sample was determined from the difference between the sample weight and the weight of the packaging and glass cloth. The ratio of hardener to resin was calculated based upon the amount of hardener as determined from the absorbance at 454 nm.

3. RESULTS AND DISCUSSION

The first quantitative studies that we conducted were directed toward determining the best design for the mixing chamber of the repair kit. These initial studies showed that the main problems with mixing were related to the disparity between the viscosities and volumes of the two components of the resin system. The viscosity of Curing Agent U more nearly matches the viscosity of Epon 828 than does the viscosity of DETA. Also, because the amine equivalent weight of Curing Agent U is higher than that of DETA, the volume ratio of Curing Agent U to Epon 828 is higher than the volume ratio of DETA to Epon 828. Because of these advantages, we also conducted studies with Epon 828 resin and Curing Agent U.

After we had completed the mixing-chamber studies, we investigated the effect of the design of the liquid-components reservoir on the thoroughness of mixing. Other resin/hardener systems were then considered using the reservoir and mixing-chamber designs that performed best in the early studies. Finally, we considered processing conditions and their effect on mixing. The results of these tests are described in the following sections.

In fabricating the kits for testing, we found that the amounts of the two epoxy components that were contained in each kit were somewhat difficult to control. This was because part of the liquid would be lost during the heat-sealing step. We were not overly concerned about this shortcoming because we believed that the use of automated equipment in a manufacturing operation would overcome this problem.

3.1 Study of Mixing-Chamber Design.

Thirty kits were fabricated in the general pattern of Figure 1. All of these had the five-section liquid-components reservoir shown in Figure 2.C. We prepared 15 of the kits using DETA as the hardener and 15 using Curing Agent U as the hardener. The kits were processed as described in Section 2.5.2. A single pass through the processor at a linear velocity of 4.4 ft/min was used for all of the kits. The results of these experiments are presented in Tables 1 and 2. For each kit, the initial composition of the resin and hardener are given. The design of the mixing chamber is described by referencing the appropriate diagram in Figure 3. The length of each pattern is also given. The mixing data give the actual weight percent of hardener found in each sample as well as the mean and standard deviation, S, for the kit. As shown, none of the kits prepared with DETA provided good mixing. Similar poor results were observed for Curing Agent U. Although some improvement was noted for kits C814-93-B-1 and -2, these results are not conclusive because of the low amount of hardener in the processed mixture compared to the initial ratio of resin to hardener in the kit.

TABLE 1. EFFECT OF MIXING-CHAMBER DESIGN ON MIXING OF EPON 828/DETA SYSTEM

Kit CB14-76	Initial composition		Mixing chamber		Mixing data			
	Epon 828, wt %	DETA, wt %	Design	Length, in.	Sample	Hardener, wt %	Mean, wt %	S
-A-1	90.3	9.7	3.A	5	A	6.4	8.9	5.4
					B	15.0		
					C	5.3		
-A-2	90.8	9.2	3.A	5	A	15.0	10.0	4.6
					B	5.5		
					C	10.0		
-A-3	90.6	9.4	3.A	5	A	3.8	9.3	10.0
					B	3.1		
					C	21.0		
-C-1	94.7	5.3	3.A	9	A	12.0	9.5	4.5
					B	4.4		
					C	12.0		
-C-2	92.9	7.1	3.A	9	A	101.0	35.0	57.0
					B	1.8		
					C	1.9		
-C-3	93.2	6.8	3.A	9	A	101.0	36.0	57.0
					B	2.0		
					C	5.2		
-B-1	90.7	9.3	3.B	4	Package ruptured			
			3.A	1				
-B-2	91.0	9.0	3.B	4	A	7.7	14.0	8.9
					B	9.1		
					C	24.0		
-B-3	91.7	8.3	3.B	4	Package ruptured			
			3.A	1				
-D-1	95.6	4.4	3.B	4	A	3.7	18.0	14.0
			3.A	5	B	33.0		
					C	18.0		
-D-2	92.9	7.1	3.B	4	A	2.5	7.0	4.9
			3.A	5	B	12.0		
					C	6.4		
-D-3	93.2	6.8	3.B	4	A	5.7	6.9	3.7
			3.A	5	B	3.9		
					C	11.0		
-E-1	93.0	7.0	3.B	8	A	5.3	5.3	1.5
			3.A	1	B	3.8		
					C	6.8		
-E-2	87.8	12.2	3.B	8	A	17.6	15.0	6.7
			3.A	1	B	7.5		
					C	20.0		
-E-3	88.2	11.8	3.B	8	Package ruptured			
			3.A	1				

TABLE 2. EFFECT OF MIXING-CHAMBER-DESIGN ON MIXING OF EPON 828/CURING AGENT U

Kit C814-93	Initial composition		Mixing chamber		Mixing data			
	Epon 828, wt %	Curing Agent U, wt %	Design	Length, in.	Sample	Hardener, wt %	Mean, wt %	S
-A-1	82.3	17.7	3.A	5	A	7.4	22.0	13.0
					B	32.0		
					C	27.0		
-A-2	78.3	21.6	3.A	5	A	3.3	21.0	19.0
					B	18.0		
					C	42.0		
-A-3	73.0	27.0	3.A	5	A	1.0	14.0	14.0
					B	30.0		
					C	12.0		
-C-1	69.2	30.8	3.A	9	A	--		
					B	12.0		
					C	11.0		
-C-2	76.6	23.4	3.A	9	Package ruptured			
-C-3	71.5	28.5	3.A	9	A	--		
					B	16.0		
					C	18.0		
-B-1	69.5	30.5	3.B	4	A	10.0	10.0	0.0
			3.A	1	B	10.0		
					C	10.0		
-B-2	72.6	27.4	3.B	4	A	10.0	11.0	0.0
			3.A	1	B	11.0		
					C	11.0		
-B-3	80.4	19.6	3.B	4	Package ruptured			
			3.A	1				
-D-1	72.3	27.7	3.B	4	Package ruptured			
			3.A	5				
-D-2	58.7	40.3	3.B	4	Package ruptured			
			3.A	5				
-D-3	70.8	29.2	3.B	4				
			3.A	5				
-E-1	72.3	27.7	3.B	8	Package ruptured			
			3.A	1				
-E-2	76.5	23.5	3.B	8	A	35.0	24.0	12.0
			3.A	1	B	25.0		
					C	10.0		
-E-3	71.6	28.4	3.B	8	A	11.0	11.0	0.0
			3.A	1	B	11.0		
					C	11.0		

3.2 Effect of Liquid-Components-Reservoir Design.

To determine the effect of the arrangement of the resin and hardener compartments on mixing, we manufactured 15 kits with different liquid-components-reservoir designs. Epon 828 and Curing Agent U were used in all of the kits. All of the kits had a 4 x 4-in. liquid-components reservoir, a 4 x 15-in. mixing chamber, a 4 x 4-in. patch compartment, and a 2 x 4-in. excess resin reservoir for an overall length of 24 in. Each kit was processed in a single pass through the rollers at a speed of 4.4 ft/min. The results of these tests are presented in Table 3. The liquid-components reservoir for each kit is described by referencing the appropriate diagram in Figure 2. The mixing chamber used in these studies was an 8-in. pattern of design 3.B followed by a 7-in. section of design 3.A. Here again, mixing was poor. It appears, however, that the more subdivided liquid-components reservoir of design 2.B improved mixing. These experiments also confirmed the difficulty of reproducing a burstable seal that would not leak.

3.3 Effect of Resin and Hardener Composition on Mixing.

We attempted to improve mixing by working with resin systems that were lower in viscosity than the Epon 828. To do this, we diluted 600 g of Epon 828 with 108 g of Araldite RD-2 Reactive Diluent. Although the viscosity of this system is much lower than neat Epon 828, it is still higher than the viscosity of neat DETA. For this reason, we prepared a mixture of 397 g of DETA and 529 g of Curing Agent U which more nearly matched the viscosity of the mixed-resin system. Fourteen kits were fabricated with the mixed-resin system. Seven of these incorporated DETA as the hardener. The DETA/Curing Agent U mixture was used as the hardener in the other seven. All of the kits were prepared with the liquid-components reservoir shown in Figure 2.B. The mixing chamber designs used in these studies are listed in Table 4 along with the results of the experiments. The kits were processed in a single pass at 4.4 ft/min. As expected, better mixing was obtained with the lower-viscosity materials. Although the viscosity of the mixed-hardener system more nearly matches the viscosity of the mixed-resin system, better mixing was obtained when neat DETA was used as the hardener. Also, a slight improvement in mixing was observed with the longer mixing chamber.

3.4 Effect of Processor Speed.

To determine the effect of the speed of the rollers during processing, five kits were fabricated and tested at speeds ranging from 1.1 to 5.5 ft/min. The liquid-components reservoir shown in Figure 2.B and the mixing chamber shown in Figure 3.D were used in each kit. The results of this study are shown in Table 5. These results show that mixing can be significantly improved by reducing the speed of the rollers.

TABLE 3. EFFECT OF LIQUID-COMPONENTS RESERVOIR ON MIXING
WITH EPON 828/CURING AGENT U SYSTEM

Kit CB14-104	Initial composition		Liquid-components reservoir design	Mixing data			
	Epox 828, wt %	Curing Agent U, wt %		Sample	Hardener, wt %	Mod, wt %	S
-D-1	85.0	15.0	2.A	Package ruptured			
-D-2	75.3	24.7	2.A	A	98.0	47.0	36.0
				B	44.0		
				C	14.0		
				D	34.0		
-D-3	69.6	30.4	2.A	A	59.0	38.0	29.0
				B	67.0		
				C	53.0		
				D	7.7		
				E	5.1		
-A-1	85.9	14.1	2.D	A	7.6	12.0	5.8
				B	21.0		
				C	12.0		
				D	10.0		
-A-2	83.9	16.1	2.D	A	35.0	23.0	12.0
				B	8.6		
				C	29.0		
				D	20.0		
				E	26.0		
-A-3	90.8	9.2	2.D	A	5.0		
				B	6.8		
-B-1	88.6	11.4	2.F	Package ruptured			
-B-2	92.6	7.4	2.E	Package ruptured			
-B-3	65.6	34.4	2.E	Package ruptured			
-C-1	82.1	17.9	2.C	A	52.0	33.0	14.0
				B	19.0		
				C	37.0		
				D	26.0		
-C-2	75.4	24.6	2.C	A	40.0	30.0	8.1
				B	30.0		
				C	36.0		
				D	22.0		
				E	22.0		
-C-3	80.6	19.4	2.C	A	23.0	20.0	4.0
				B	16.0		
				C	16.0		
				D	20.0		
				E	24.0		

TABLE 4. EFFECT OF RESIN AND HARDENER COMPOSITIONS ON MIXING

Kit CB14-122	Kit contents				Mixing chamber		Mixing data		
	Epon 828, wt %	Araldite RD2, wt %	DETA, wt %	Curing Agent U, wt %	Design	Length, in.	Number of samples	Mean hardener, wt %	S
-A-1	58.6	10.6	30.8	--	3.C	8	3	39.0	8.7
-A-2	57.8	10.4	31.8	--	3.C	8	4	28.0	8.7
-A-3	57.9	10.4	31.7	--	3.C	8	5	40.0	8.4
-A-4	58.9	10.6	30.4	--	3.C	8	5	32.0	4.1
-A-5	60.8	10.9	28.2	--	3.C	8	5	25.0	7.1
-B-4	58.1	10.5	31.4	--	3.D	12	Package ruptured		
-B-5	55.8	10.2	33.0	--	3.D	12	Package ruptured		
-C-1	48.4	8.7	37.7	5.2	3.C	8	5	50.0	21.0
-C-2	48.8	8.8	37.2	5.1	3.C	8	5	40.0	12.0
-C-3	49.2	8.9	36.9	5.0	3.C	8	8	34.0	24.0
-C-4	48.6	8.7	37.6	5.1	3.C	8	5	34.0	26.0
-B-1	50.0	8.8	37.1	5.1	3.D	12	Package ruptured		
-B-2	45.7	8.2	40.6	5.5	3.D	12	5	50.0	16.0
-B-3	52.2	9.4	33.7	4.6	3.D	12	4	51.0	17.0

TABLE 5. EFFECT OF PROCESSOR SPEED ON MIXING

Kit CB14-122	Kit contents				Processor	Mixing data		
	Epon 828, wt %	Araldite RD-2, wt %	DETA, wt %	Curing Agent U, wt %	Linear velocity, ft/min	Number of samples	Mean hardener, wt %	S
-D-1	46.9	8.5	39.2	5.4	5.5	5	56.0	30.0
-D-2	48.4	8.7	37.7	5.2	4.4	5	37.0	8.2
-D-3	47.8	8.6	38.4	5.2	3.3	4	51.0	4.2
-D-4	40.9	7.4	45.5	6.2	2.2	4	44.0	5.1
-D-5	45.3	8.2	40.9	5.6	1.1	5	56.0	6.4

3.5 Effect of Multiple Passes on Mixing.

Ten kits were fabricated with a 14-in. mixing chamber of design 3.A. The liquid-components reservoir in these kits was design 2.B. Each kit was processed at a speed of 4.4 ft/min, and either one, three, or five passes through the processor were used. As shown in Table 6, mixing is improved as the number of passes is increased.

3.6 Fibrous Pad Containing Hardener.

We next investigated an alternative design for the mixing chamber that would provide a more tortuous path than any of the designs discussed thus far. The design incorporated a fibrous pad in the mixing chamber. The pad is impregnated with the hardener, leaving only the resin mixture in the liquid-components reservoir. In our first experiments using this design, the

spectroscopy results indicated that the mixed resin contained more than 100% hardener. Apparently the DETA extracted a soluble component from the pads that interfered with the spectroscopic method. Visual inspection of the mixed resin indicated that mixing was relatively uniform.

3.7 Effect of Mixing With a Hand Roller.

The first kit design that we considered included a two-compartment reservoir and a patch compartment. The two-compartment reservoir was connected by a burstable seal, and mixing was accomplished in the reservoir with a hand-held roller. Six kits were prepared and evaluated to determine the effectiveness of hand mixing. As shown in Table 7, mixing improves as the number of passes of the roller across the kit increases.

TABLE 6. EFFECT OF NUMBER OF PASSES THROUGH PROCESSOR ON MIXING

Kit (814-134)	Kit contents				Number of passes	Mixing data		
	Epon 828 wt %	Araldite RD2, wt %	DETA wt %	Curing Agent U, wt %		Number of samples	Mean hardener, wt %	S
-A-1	57.4	10.4	28.3	3.9	1	5	31.0	20.0
-A-2	54.0	9.7	31.9	4.4	1	5	54.0	22.0
-A-3	52.8	9.5	33.2	4.5	1	5	55.0	24.0
-A-4	50.6	9.1	35.5	4.8	1	5	58.0	27.0
-A-5	54.5	9.8	31.4	4.3	3	5	38.0	11.0
-B-1	50.9	9.2	35.1	49.3	3	5	48.0	13.0
-B-2	53.9	9.6	32.2	4.3	Package ruptured			
-B-3	51.1	9.2	35.0	4.7	5	5	44.0	3.1
-B-4	57.2	10.3	28.6	3.9	5	5	33.0	6.5
-B-5	53.3	9.6	32.6	4.5	5	5	36.0	6.4

TABLE 7. EFFECT OF HAND PROCESSING ON MIXING

Kit	Epon 828, wt %	DETA wt %	Number of passes	Number of samples	Mean hardener, wt %	S
-1	57.0	43.0	5	4	34.0	10.0
-2	63.2	36.8	15	5	38.0	11.0
-3	78.2	21.8	15	5	20.0	5.0
-4	68.6	31.4	50	4	27.0	1.6
-5	74.8	25.2	50	5	22.0	2.1
-6	74.4	25.6	50	5	21.0	1.7

3.8 Pot-Life Studies.

A study to determine the pot lives and cure times (time to hardness) was conducted using different stoichiometric ratios of hardener to resin. The components were mixed by hand in polyethylene beakers, and the elapsed time before they gelled (pot life) and completely hardened (cure time) were determined. This procedure was performed with a mixture of 15.28% by weight of Araldite RD-2 Reactive Diluent and 84.72% Epon 828 as the resin. DETA was used as the hardener in one study, and a mixture of 88.25% DETA and 11.75% Curing Agent U was used in the other. The results are shown in Figures 4 and 5 respectively. In general, both systems showed a wide tolerance for the resin-to-hardener ratio in terms of the pot life and cure time. The effect of the ratio on the strength of the cured resin was not determined.

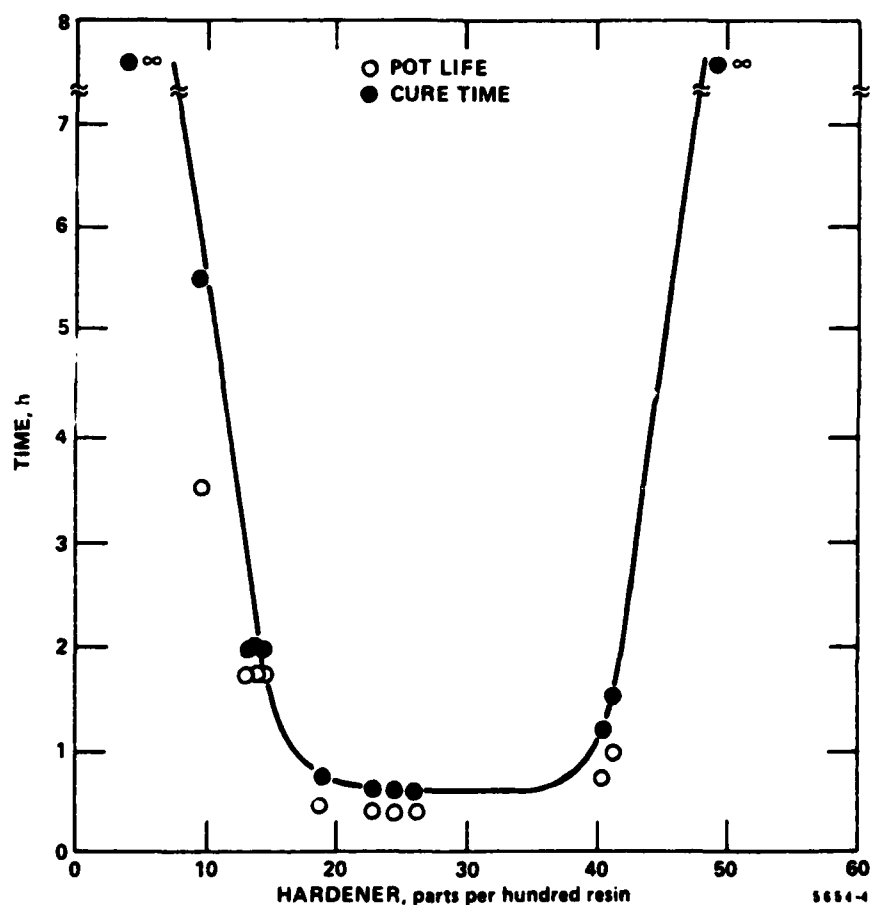


Figure 4. Pot life and cure time for Epon 828: RD-2/DETA System.

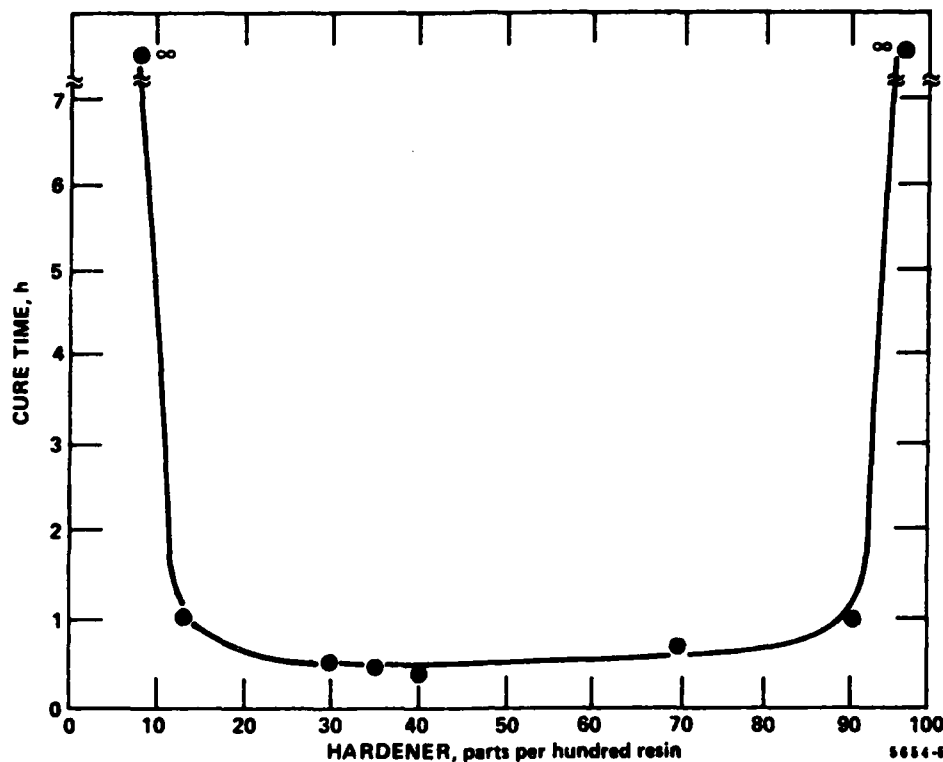


Figure 5. Pot life for Epon 828: Araldite RD-2/DETA: Curing Agent U System.

4. CONCLUSIONS AND RECOMMENDATIONS

The results obtained from the mixing studies show that mixing can be influenced by the design of the liquid-components reservoir and the mixing chamber. Other factors, however, such as the viscosity of the liquid components, the speed at which the kit is processed, and the number of passes through the processor, have a greater influence on the degree of mixing that can be obtained. The best mixing that we obtained was achieved with a hand-held roller of the type sold by Tra-Con, Medford, MA. In 50 passes with the hand-held roller, we obtained better mixing than in any of the experiments involving the wringer-type processor.

We believe that a kit based on the use of a hand-held roller can be developed most rapidly and that the kit can accommodate a wide range of resin types. However, because a kit based on a hand-held roller would not represent a significant advancement over commercially available products, we do not recommend that this approach be pursued further. While we have not yet demonstrated thorough mixing with a wringer-type processor, we feel that an acceptable kit can be developed for certain low-viscosity systems.

5. ACKNOWLEDGMENT

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