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DEVELOPMENT OF BATTERY SEPARATOR MATERIAL PROCESS

by

L. M. Adams
W. W. Harlowe, Jr.

QUARTERLY REPORT

SwRI Project No. 01-2015
Contract No. 951718

Prepared for

Jet Propulsion Laboratory
California Institute of Technology
4800 Oak Grove Drive
Pasadena, California 91103

Attn: H. E. Patterson
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Mail Station 190-212

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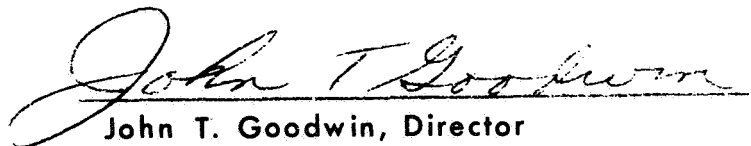
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ABSTRACT

The poly(acrylic acid) content of acrylic acid-grafted polyethylene film is proportional to the acrylic acid content of the grafting solution used to graft the polyethylene film. The electrical resistance of the grafted film decreases as the poly(acrylic acid) content of the grafted film increases. The electrical resistance also decreases when the temperature is increased in the neutralizing and rinsing bath. However, the higher temperature may be detrimental to the product as the silver ion migration increases when the neutralizing and rinsing temperature is increased.

Properties of battery separator material prepared in large quantities are presented.

Methacrylic acid-grafted polyethylene film has low electrical resistance, high tensile strength, and high elongation. This material decreases in length from the dry dimension when sterilized in 40 percent potassium hydroxide.

Polyethylene film grafted with mixtures of acrylic acid and methacrylic acid are being evaluated.

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I. SUMMARY

The poly(acrylic acid) content of acrylic acid-grafted polyethylene film is proportional to the acrylic acid content of the grafting solution used to graft the polyethylene film. The electrical resistance of the grafted film decreases as the poly(acrylic acid) content of the grafted film increases. The electrical resistance also decreases when the temperature is increased in the neutralizing and rinsing baths. However, the higher temperature may be detrimental to the product as the silver ion migration increases when the neutralizing and rinsing temperature is increased.

Properties of battery separator material prepared in large quantities are presented.

Methacrylic acid-grafted polyethylene film has low electrical resistance, high tensile strength, and high elongation. This material decreases in length from the dry dimension when sterilized in 40 percent potassium hydroxide.

Polyethylene film grafted with mixtures of acrylic acid and methacrylic acid are being evaluated.

II. INTRODUCTION

The change in polyethylene film used in the preparation of battery separator material has made it necessary to redetermine optimum processing conditions, as this material behaves differently than the previously used polyethylene film. Efforts during this quarter have been directed toward finding the optimum processing conditions. Other monomers for grafting polyethylene were also investigated.

This report covers work performed during the period from April 1, 1969 through July 1, 1969.

III. EXPERIMENTAL

A. Effect of Acrylic Acid Concentration

The effect of acrylic acid concentration in the grafting solution has been discussed in previous reports. The data presented were for films which had been grafted only, and comparisons of only the resistance were made. Some of these grafted films have been crosslinked and then sterilized (Table 1).

On comparison with the electrical resistances of the grafted film before and after crosslinking, the crosslinking increases the resistance slightly in most cases. Neutralization and washing at 97°C produces films having lower resistances than those processed at 80°C.

The film thickness and change in thickness on wetting with 40 percent potassium hydroxide change little if any on decreasing the concentration of acrylic acid in the grafting solution. However, the increase in width from the dry dimension on wetting and sterilization tends to become smaller as the acrylic acid concentration decreases. The same is true for changes in length, but at the lower concentrations of acrylic acid, there is a decrease in length from the dry dimension on sterilization in 40 percent potassium hydroxide.

The effect on composition of the grafted polyethylene film has been determined from a series of runs in which the acrylic acid concentration of the grafting solution was varied from 10 to 25 weight percent.

The weight ratio of acrylic acid to chain terminator (carbon tetrachloride) was maintained at 5 to 1.

During the process of rolling the 25-foot rolls of polyethylene film, small sections of film were cut from the roll at five-foot intervals, weighed, and replaced in their respective places in the roll. The rolls were then grafted and processed in the usual manner.

The weighed sections were converted back to the free acid form and dried in a vacuum desiccator to a constant weight. From the weight increase of the sections, the degree of grafting and the weight percent poly(acrylic acid) graft were calculated. The degree of grafting varied with the concentration of acrylic acid in the grafting solution (Table 2) and was quite uniform throughout each roll. The degree of grafting occurring with change in acrylic acid concentration is also shown in Figure 1.

The electrical resistance was uniform throughout each roll, and as expected, it decreased with increasing acrylic acid concentration (Table 3 and Figure 2).

B. Material Prepared in Large Reactors

The electrical resistances of grafted and crosslinked polyethylene film which was prepared in 100-foot rolls and shipped to the Jet Propulsion Laboratory are presented in Tables 4-12.

Analytical data are presented in Table 13 for four rolls of grafted film. Erratic electrical resistances were obtained after

sterilization of part of Sample No. 3-358. The outer portion of the roll was uniform, but the inner portion (next to rod) was erratic. No explanation of this erratic behavior is available at the present time.

C. Effect of Chain Terminator Concentration

The effect of the concentration of chain terminator in the grafting solution on the electrical resistance of the polyethylene film (grafted only) is slight over the range evaluated (Table 14). However, from previous experience, gross differences are expected when the grafted film is crosslinked and then sterilized. Analytical data will be presented in the next quarterly report.

D. Other Grafting Systems

A study of the use of mixtures of acrylic acid and methacrylic acid for grafting polyethylene film has been initiated. Preliminary data are presented in Tables 15-17.

The effect of concentration of methacrylic acid in the grafting solution on the grafting of polyethylene film (grafted only) was reported in the April 10, 1969 Quarterly Report. This series has been crosslinked and analyzed (Table 18). The effect of neutralization and rinsing temperature on the methacrylic acid-grafted films is similar to the effect obtained with acrylic acid-grafted films in that lower electrical resistances are obtained before sterilization with the higher processing temperatures. This effect is nullified by sterilization. The methacrylic acid-grafted films have high tensile strength and elongation.

E. Silver Migration Studies

Preliminary data on silver migration through acrylic acid-grafted membranes indicate:

1. Silver ion migration rate decreases with a decrease in the degree of grafting.
2. Silver migration rate decreases when high concentrations of chain terminator (carbon tetrachloride) are used in the grafting solution.
3. Silver migration rate is much lower for acrylic acid-grafted films neutralized and rinsed at 80°C than for the acrylic acid-grafted films neutralized and rinsed at 97°C.

Data for silver-ion migration will be reported when complete analyses of the grafted films are obtained.

IV. CONCLUSIONS

Increasing the acrylic acid content of the grafting solution increases the poly(acrylic acid) content of the grafted polyethylene film with a resultant decrease in electrical resistance and an increase in the rate of silver migration.

Increasing the temperature of the neutralizing and rinsing baths from 80° to 97°C decreases the resistance of the battery separator material and increases the rate of silver migration.

Preliminary data for polyethylene grafted with methacrylic acid and acrylic acid-methacrylic acid mixtures are encouraging.

TABLE 1. SAMPLE NOS. 330, 332, 333, AND 335
GRAFTED AND CROSSLIKED

Grafting Solution Composition

<u>330</u>	<u>332</u>	<u>333</u>	<u>335</u>
25	20	15	10 wt % Acrylic acid
70	76	82	88 wt % Benzene
5	4	3	2 wt % Carbon tetrachloride

Experimental Conditions for Grafting

Dose Rate:	0.012 Mrad/hr
Total Dose:	0.815 Mrad
Temperature:	77°F
Atmosphere:	Nitrogen
Roll Length:	30 feet

A. Electrical Properties

<u>Sample No.</u>	<u>Average Resistance,</u> <u>milliohm-inch²</u>		<u>Standard Deviation</u>		<u>Standard Deviation</u> <u>% of Average</u>	
	<u>B.S. (1)</u>	<u>A.S. (2)</u>	<u>B.S. (1)</u>	<u>A.S. (2)</u>	<u>B.S. (1)</u>	<u>A.S. (2)</u>
330 ⁽³⁾	12	7	3.0	0.6	25.7	8.6
330 ⁽⁴⁾	7	6	0.6	0.5	9.2	8.0
332 ⁽³⁾	12	10	1.1	2.1	9.3	20.5
332 ⁽⁴⁾	10	8	1.7	1.2	16.7	13.8
333 ⁽³⁾	24	18	3.0	3.1	12.3	17.8
333 ⁽⁴⁾	16	16	1.6	2.0	9.9	12.7
335 ⁽³⁾	33	85	8.5	58.5	26.0	69.0
335 ⁽⁴⁾	22	38	1.6	6.5	7.4	16.9

TABLE 1 (Continued)

B. Physical Properties

Sample No.	Thickness, mil			Dimensional Changes ⁽⁵⁾ , %				Tensile Strength ⁽¹⁾ , psi	Elongation ⁽¹⁾ , %
	Dry	B. S. ⁽¹⁾	A. S. ⁽²⁾	B. S. ⁽¹⁾	A. S. ⁽²⁾	B. S. ⁽¹⁾	A. S. ⁽²⁾		
330 ⁽³⁾	1.1	1.4	1.5	10.0	10.0	8.8	4.4	1026	80
330 ⁽⁴⁾	1.1	1.3	1.5	10.0	8.0	8.8	2.5	975	89
332 ⁽³⁾	1.2	1.4	1.6	9.0	7.0	8.9	3.0	1000	75
332 ⁽⁴⁾	1.1	1.3	1.5	11.0	10.0	8.4	1.5	1093	87
333 ⁽³⁾	1.2	1.3	1.5	7.0	6.0	6.4	-0.5	1445	95
333 ⁽⁴⁾	1.2	1.3	1.5	10.0	9.0	6.9	-0.5	1334	95
335 ⁽³⁾	1.1	1.2	1.7	5.9	2.9	5.4	-3.5	1514	80
335 ⁽⁴⁾	1.1	1.2	1.6	8.0	6.0	6.4	-1.0	1351	83

(1) Before sterilization - wet with 40% KOH.

(2) After sterilization - wet with 40% KOH.

(3) Neutralized and rinsed at 80°C.

(4) Neutralized and rinsed at 97°C.

(5) Change from dry dimension.

TABLE 2. EFFECT OF ACRYLIC ACID CONCENTRATION IN GRAFTING SOLUTION ON COMPOSITION OF THE GRAFTED FILM

Sample No.	Grafting Solution Composition, wt %			Footage	Degree of Grafting ⁽¹⁾	Poly(acrylic acid) in Product, wt %	Poly(acrylic acid), meq ⁽²⁾ /gram Product
	Acrylic Acid	CCl ₄	C ₆ H ₆				
S-81	25	5	75	5	0.85	45.6	6.25
				10	0.86	46.2	6.32
				15	0.89	47.2	6.46
				20	0.86	46.4	6.36
S-82	20	4	76	5	0.75	42.9	5.88
				10	0.69	40.9	5.60
				15	0.64	39.1	5.36
				20	0.71	41.7	5.71
S-83	15	3	82	5	0.51	33.9	4.64
				10	0.50	33.1	4.53
				15	0.51	33.7	4.61
				20	0.54	34.9	4.78
S-84	10	2	88	5	0.40	28.6	3.92
				10	0.37	27.0	3.70
				15	0.37	27.1	3.71
				20	0.38	27.4	3.75

(1) Weight of poly(acrylic acid) in grafted polyethylene film/weight of starting polyethylene film.

(2) Theoretical milliequivalent.

TABLE 3. SAMPLE NOS. S-81 - S-84
GRAFTED ONLY

<u>Grafting Solution Composition</u>				<u>Experimental Conditions for Grafting</u>	
<u>S-81</u>	<u>S-82</u>	<u>S-83</u>	<u>S-84</u>	Dose Rate:	0.012 Mrad/hr
25	20	15	10 wt % Acrylic acid	Total Dose:	0.815 Mrad
70	76	82	88 wt % Benzene	Temperature:	79 °F
5	4	3	2 wt % CCl ₄	Atmosphere:	Nitrogen
				Roll Length:	25 feet

Electrical Properties

<u>Footage</u>	<u>Resistance, milliohm-inch²</u>			
	<u>S-81</u>	<u>S-82</u>	<u>S-83</u>	<u>S-84</u>
5	7	9	12	14
10	7	9	13	14
15	7	10	11	16
20	6	9	10	16

Exotherm Data

Time to exotherm, hr:	12.0	12.0	12.0	12.0
Time to max. exotherm, hr:	20.0	20.0	20.0	20.0
Maximum temperature, °F:	82	81	81	82

Note

Prepared from Dow 400 (1 mil) polyethylene film
with Chicopee No. 44 cheesecloth interlayer.
Neutralized and rinsed at 97°C.

TABLE 5. SAMPLE NUMBER 358GX

<u>Grafting Solution Composition</u>		<u>R e s u l t s</u>	
15	wt % Acrylic acid		
82	wt % Benzene	<u>Footage</u>	<u>Resistance, milliohm-inch²</u>
3	wt % Carbon tetrachloride		
<u>Crosslinking Solution Composition</u>			
1.0	vol % Divinylbenzene	1-358-3	10
1.0	vol % Benzene		11
98.0	vol % Methanol	1-358-100	16
<u>Experimental Conditions for Grafting</u>			16
Dose rate:	0.012 Mrad/hr	2-358-3	11
Total dose:	0.815 Mrad		9
Temperature:	59°F	2-358-100	14
Atmosphere:	Nitrogen		15
Roll length:	100 feet (5 rolls per reactor)	3-358-3	12
<u>Experimental Conditions for Crosslinking</u>			11
Dose rate:	0.025 Mrad/hr	3-358-100	16
Total dose:	0.550 Mrad		17
Temperature:	75°F	4-358-3	15
Atmosphere:	Nitrogen		12
<u>Exotherm During Grafting</u>		4-358-60	19
Time to exotherm, hr:	6		17
Time to maximum exotherm, hr:	15	4-358-100	16
Maximum temperature, °F:	161		18
<u>Neutralization and Washing Temperature</u>		5-358-3	16
97°C			18
<u>Remarks</u>		5-358-100	13
Dow 400 (1 mil)-Chicopee No. 44 interlayer.			8
Footage shipped: 530 feet			

TABLE 7. SAMPLE NUMBER 359GX

Grafting Solution Composition

15 wt % Acrylic acid
 82 wt % Benzene
 3 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
 1.0 vol % Benzene
 98.0 vol % Methanol

Experimental Conditions for Grafting

Dose rate: 0.012 Mrad/hr
 Total dose: 0.815 Mrad
 Temperature: 59°F
 Atmosphere: Nitrogen
 Roll length: 100 feet (5 rolls/reactor)

Experimental Conditions for Crosslinking

Dose rate: 0.025 Mrad/hr
 Total dose: 0.550 Mrad
 Temperature: Ambient
 Atmosphere: Nitrogen

Exotherm During Grafting

Time to exotherm, hr: 6
 Time to maximum exotherm, hr: 15
 Maximum temperature, °F: 113

Neutralization and Washing Temperature

97°C

Remarks

Dow 400 (1-mil)-Chicopee No. 44
 interlayer.

Footage shipped: 300 feet

R e s u l t s

<u>Footage</u>	<u>Resistance, milliohm-inch²</u>
3-359-4	13
	16
3-359-110	10
	15
2-359-4	10
	8
2-359-110	20
	20
1-359-4	12
	14
1-359-65	12
	12
1-359-110	14
	12

TABLE 9. SAMPLE NUMBER 361GX

<u>Grafting Solution Composition</u>		<u>R e s u l t s</u>	
15	wt % Acrylic acid		
82	wt % Benzene	<u>Footage</u>	<u>Resistance,</u> <u>milliohm-inch²</u>
3	wt % Carbon tetrachloride		
<u>Crosslinking Solution Composition</u>		5-361-4	18
1.0	vol % Divinylbenzene		19
1.0	vol % Benzene	5-361-104	17
98.0	vol % Methanol		14
<u>Experimental Conditions for Grafting</u>		4-361-4	13
Dose rate:	0.012 Mrad/hr		13
Total dose:	0.815 Mrad	4-361-108	10
Temperature:	73°F		12
Atmosphere:	Nitrogen	3-361-4	14
Roll length:	100 feet (5 rolls/reactor)		11
<u>Experimental Conditions for Crosslinking</u>		3-361-106	11
Dose rate:	0.025 Mrad/hr		12
Total dose:	0.550 Mrad	2-361-4	15
Temperature:	Ambient		16
Atmosphere:	Nitrogen		17
<u>Exotherm During Grafting</u>		2-361-112	13
Time to exotherm, hr:	13		20
Time to maximum exotherm, hr:	23	1-361-4	21
Maximum temperature, °F:	111		10
<u>Neutralization and Washing Temperature</u>		1-361-52	11
97°C			8
<u>Note</u>		1-361-96	8
Dow 400 (1 mil)-Chicopee No. 44			
interlayer.			
Footage shipped: 491			

TABLE 10. SAMPLE NUMBER 362GX

<u>Grafting Solution Composition</u>		<u>R e s u l t s</u>	
15 wt % Acrylic acid			
82 wt % Benzene		<u>Footage</u>	<u>Resistance, milliohm-inch²</u>
3 wt % Carbon tetrachloride			
<u>Crosslinking Solution Composition</u>			
1.0 vol % Divinylbenzene		1-362-6	18
1.0 vol % Benzene			12
98.0 vol % Methanol		1-362-125	14
<u>Experimental Conditions for Grafting</u>			
Dose rate:	0.012 Mrad/hr		9
Total dose:	0.815 Mrad	2-362-6	12
Temperature:	64°F		8
Atmosphere:	Nitrogen	2-362-124	12
Roll length:	110 feet (4 rolls/reactor)		12
<u>Experimental Conditions for Crosslinking</u>		3-362-6	17
Dose rate:	0.025 Mrad/hr		13
Total dose:	0.550 Mrad	3-362-122	8
Temperature:	Ambient		10
Atmosphere:	Nitrogen	4-362-6	20
<u>Exotherm During Grafting</u>			17
Time to exotherm, hr:	4.0	4-362-122	11
Time to maximum exotherm, hr:	20.0		12
Maximum temperature, °F:	111		
<u>Neutralization and Washing Temperature</u>			
97°C			
<u>Note</u>			
Dow 400 (1 mil)-Chicopee			
No. 44 interlayer			
Footage shipped: 469			

TABLE 11. SAMPLE NUMBER 363GX

<u>Grafting Solution Composition</u>		<u>R e s u l t s</u>	
15	wt % Acrylic acid		
82	wt % Benzene	<u>Footage</u>	<u>Resistance,</u> <u>milliohm-inch²</u>
3	wt % Carbon tetrachloride		
<u>Crosslinking Solution Composition</u>		1-363-6	10
			9
1.0	vol % Divinylbenzene	1-363-122	9
1.0	vol % Benzene		10
98.0	vol % Methanol	2-363-6	13
<u>Experimental Conditions for Grafting</u>			9
		2-363-1	11
			17
Dose rate:	0.012 Mrad/hr	3-363-6	18
Total dose:	0.815 Mrad		14
Temperature:	64°F	3-363-118	10
Atmosphere:	Nitrogen		10
Roll length:	110 feet (4 rolls/reactor)	4-363-6	11
<u>Experimental Conditions for Crosslinking</u>			9
		4-363-123	10
			7
Dose rate:	0.025 Mrad/hr		
Total dose:	0.550 Mrad		
Temperature:	Ambient		
Atmosphere:	Nitrogen		
<u>Exotherm During Grafting</u>			
Time to exotherm, hr:	4.0		
Time to maximum exotherm, hr:	20.0		
Maximum temperature, °F:	100		
<u>Neutralization and Washing Temperature</u>			
97°C			
<u>Note</u>			
Dow 400 (1 mil)-Chicopee			
No. 44 interlayer.			
Footage shipped: 450			

TABLE 12. SAMPLE NUMBER 375GX

Grafting Solution Composition

15 wt % Acrylic acid
 82 wt % Benzene
 3 wt % Carbon tetrachloride

Crosslinking Solution Composition

1.0 vol % Divinylbenzene
 1.0 vol % Benzene
 98.0 vol % Methanol

Experimental Conditions for Grafting

Dose rate: 0.012 Mrad/hr
 Total dose: 0.815 Mrad
 Temperature: 66°F
 Atmosphere: Nitrogen
 Roll length: 100 feet (5 rolls/reactor)

Experimental Conditions for Crosslinking

Dose rate: 0.025 Mrad/hr
 Total dose: 0.550 Mrad
 Temperature: Ambient
 Atmosphere: Nitrogen

Exotherm During Grafting

Time to exotherm, hr: 7.0
 Time to maximum exotherm, hr: 20.0
 Maximum temperature, °F: 131

Neutralization and Washing Temperature

97°C

Note

Dow 400 (1 mil)-Chicopee No. 44
 interlayer.

Footage shipped: 291 feet

R e s u l t s

<u>Footage</u>	<u>Resistance, milliohm-inch²</u>
3-375-5	18
3-375-100	8
4-375-5	12
4-375-100	9
5-375-5	11
5-375-100	11

TABLE 13. SAMPLE NOS. 2-358 - 5-358
GRAFTED AND CROSSLINKED

<u>Grafting Solution Composition</u>				<u>Experimental Conditions for Grafting</u>					
15 wt % Acrylic acid				Dose Rate:	0.012 Mrad/hr				
82 wt % Benzene				Total Dose:	0.815 Mrad				
3 wt % Carbon tetrachloride				Temperature:	59°F				
				Atmosphere:	Nitrogen				
				Roll Length:	100 feet (5 rolls per reactor)				
A. <u>Electrical Properties</u>									
Sample No.	Average Resistance, milliohm-inch ²		Standard Deviation		Standard Deviation % of Average				
	B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)			
2-358	13	12	3.6	3.3	28.2	28.2			
3-358	13	25	1.7	26.1	12.7	105.3			
4-358	13	13	1.7	1.4	12.6	10.9			
5-358	14	16	3.8	6.5	27.3	41.7			
B. <u>Physical Properties</u>									
Sample No.	Average Thickness, mil			Dimensional Changes ⁽³⁾ , %				Tensile Strength ⁽¹⁾ ,	Elongation ⁽¹⁾
	Dry			Width		Length		psi	%
		B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)		
2-358	1.1	1.3	1.6	13.0	14.0	9.5	5.5	1286	>100
3-358	1.1	1.4	1.5	13.0	14.0	9.0	3.0	1270	>100
4-358	1.2	1.4	1.7	12.0	15.0	9.5	4.0	1398	>100
5-358	1.2	1.4	1.7	15.0	16.0	10.5	5.0	1302	>100

Note: Prepared from Dow 400 (1 mil) polyethylene film with Chicopee No. 44 cheesecloth interlayer. Neutralized and rinsed at 97°C.

(1) Before sterilization - wet with 40% KOH.

(2) After sterilization - wet with 40% KOH.

(3) Change from dry dimension

TABLE 14. SAMPLE NOS. 348-352
GRAFTED ONLY

<u>Grafting Solution Composition</u>					<u>Experimental Conditions for Grafting</u>	
<u>348</u>	<u>349</u>	<u>350</u>	<u>351</u>	<u>352</u>	Dose Rate:	0.012 Mrad/hr
15	15	15	15	15	wt % Acrylic acid	Total Dose: 0.815 Mrad
82	81.25	80	77.5	70	wt % Benzene	Temperature: 75°F
3	3.75	5	7.5	15	wt % CCl ₄	Atmosphere: Nitrogen
						Roll Length: 25 feet

Electrical Properties

<u>Footage</u>	<u>Resistance, milliohm-inch²</u>				
	<u>348</u>	<u>349</u>	<u>350</u>	<u>351</u>	<u>352</u>
5 ⁽¹⁾	18, 15	18, 16	34, 19	20, 23	22, 27
15 ⁽¹⁾	14, 18	19, 22	19, 22	18, 27	22, 22
20 ⁽²⁾	9, 10	13, 12	11, 13	12, 17	15, 15
25 ⁽²⁾	12, 11	10, 10	11, 12	14, 11	14, 16

Exotherm Data

Time to exotherm, hr:	4.0	4.0	4.0	6.5	4.0
Time to maximum exotherm, hr:	14.0	14.0	14.0	10.0	10.0
Maximum temperature, °F:	78	79	80	80	93

Note

Prepared from Dow 410 (1 mil) polyethylene film
with cheesecloth interlayer.

-
- (1) Neutralized and rinsed at 80°C.
(2) Neutralized and rinsed at 97°C.

TABLE 15. SAMPLE NO. 343
GRAFTED ONLY

<u>Grafting Solution Composition</u>	<u>Experimental Conditions for Grafting</u>	
12.5 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
12.5 wt % Methacrylic acid	Total Dose:	0.815 Mrad
75.0 wt % Benzene	Temperature:	72°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

A. Electrical Properties

<u>Sample No.</u>	<u>Resistance, milliohm-inch²</u>
343-5 ⁽¹⁾	15
	14
343-15 ⁽¹⁾	12
	13
343-20 ⁽²⁾	12
	13
343-25 ⁽²⁾	14
	13

Exotherm Data

Time to exotherm, hr:	2.0
Time to maximum exotherm, hr:	4.0
Maximum temperature, °F:	76

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and washed at 80°C.

(2) Neutralized and washed at 97°C.

TABLE 16. SAMPLE NO. 353
GRAFTED ONLY

<u>Grafting Solution Composition</u>	<u>Experimental Conditions for Grafting</u>	
8.2 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
16.8 wt % Methacrylic acid	Total Dose:	0.815 Mrad
75.0 wt % Benzene	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

A. Electrical Properties

<u>Sample No.</u>	<u>Resistance, milliohm-inch²</u>
353-5 ⁽¹⁾	8
	7
353-15 ⁽¹⁾	9
	9
353-20 ⁽²⁾	9
	9
353-25 ⁽²⁾	9
	9

Exotherm Data

Time to exotherm, hr:	4.0
Time to maximum exotherm, hr:	14.0
Maximum temperature, °F:	78

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and washed at 80°C.

(2) Neutralized and washed at 97°C.

TABLE 17. SAMPLE NO. 354
GRAFTED ONLY

<u>Grafting Solution Composition</u>	<u>Experimental Conditions for Grafting</u>	
16.8 wt % Acrylic acid	Dose Rate:	0.012 Mrad/hr
8.2 wt % Methacrylic acid	Total Dose:	0.815 Mrad
75.0 wt % Benzene	Temperature:	75°F
	Atmosphere:	Nitrogen
	Roll Length:	25 feet

A. Electrical Properties

<u>Sample No.</u>	<u>Resistance, milliohm-inch²</u>
354-5 ⁽¹⁾	11
	11
354-13 ⁽¹⁾	9
	9
354-20 ⁽²⁾	9
	9
354-25 ⁽²⁾	11
	12

Exotherm Data

Time to exotherm, hr:	4.0
Time to maximum exotherm, hr:	14.0
Maximum temperature, °F:	78

Note

Prepared from Dow 400 (1 mil) polyethylene film with cheesecloth interlayer.

(1) Neutralized and washed at 80°C.

(2) Neutralized and washed at 97°C.

**TABLE 18. SAMPLE NOS. 339-342
GRAFTED AND CROSSLINKED**

<u>Grafting Solution Composition</u>				<u>Experimental Conditions for Grafting</u>	
<u>339</u>	<u>340</u>	<u>341</u>	<u>342</u>	Dose Rate:	0.012 Mrad/hr
25	20	15	10 wt % Methacrylic acid	Total Dose:	0.815 Mrad
75	80	85	90 wt % Benzene	Temperature:	72°F
				Atmosphere:	Nitrogen
				Roll Length:	25 feet

A. <u>Electrical Properties</u>						
Sample No.	Average Resistance, milliohm-inch ²		Standard Deviation		Standard Deviation % of Average	
	B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)	B. S. (1)	A. S. (2)
339 ⁽³⁾	16	6	1.5	0.4	9.7	6.7
339 ⁽⁴⁾	8	6	0.7	1.2	9.2	20.5
340 ⁽³⁾	15	7	4.3	2.0	27.6	29.2
340 ⁽⁴⁾	10	7	1.2	2.0	11.9	27.7
341 ⁽³⁾	25	12	4.5	1.2	17.9	10.1
341 ⁽⁴⁾	15	13	2.6	3.9	16.8	29.9
342 ⁽³⁾	27	10	3.3	4.8	12.2	45.5
342 ⁽⁴⁾	13	11	2.4	1.3	18.0	12.3

TABLE 18 (Continued)

B. Physical Properties

Sample No.	Average Thickness, mil			Dimensional Changes ⁽⁵⁾ , %				Tensile Strength ⁽¹⁾ , psi	Elongation ⁽¹⁾ , %
	Dry	B. S. ⁽¹⁾		Width		Length			
		A. S. ⁽²⁾	B. S. ⁽¹⁾	A. S. ⁽²⁾	B. S. ⁽¹⁾	A. S. ⁽²⁾			
339 ⁽³⁾	1.2	1.3	1.6	11.0	19.0	5.5	-4.5	1349	>100
339 ⁽⁴⁾	1.1	1.4	1.8	12.0	17.0	6.9	-5.5	1300	>100
340 ⁽³⁾	1.3	1.4	1.7	11.2	18.4	7.1	-4.0	1790	>100
340 ⁽⁴⁾	1.2	1.3	1.7	13.1	15.2	6.5	-7.5	1405	>100
341 ⁽³⁾	1.3	1.4	1.9	8.1	11.1	6.5	-5.0	1852	>100
341 ⁽⁴⁾	1.2	1.4	2.0	12.3	14.3	8.0	-6.0	1694	>100
342 ⁽³⁾	1.3	1.3	1.8	10.1	17.2	.65	-5.5	1993	>100
342 ⁽⁴⁾	1.3	1.4	1.9	11.0	13.0	7.5	-8.5	1356	>100

Note

Prepared from Dow 400 (1 mil) polyethylene film
with cheesecloth interlayer.

(1) Before sterilization - wet with 40% KOH.

(2) After sterilization - wet with 40% KOH.

(3) Neutralized and rinsed at 80°C.

(4) Neutralized and rinsed at 97°C.

(5) Change from dry dimension.

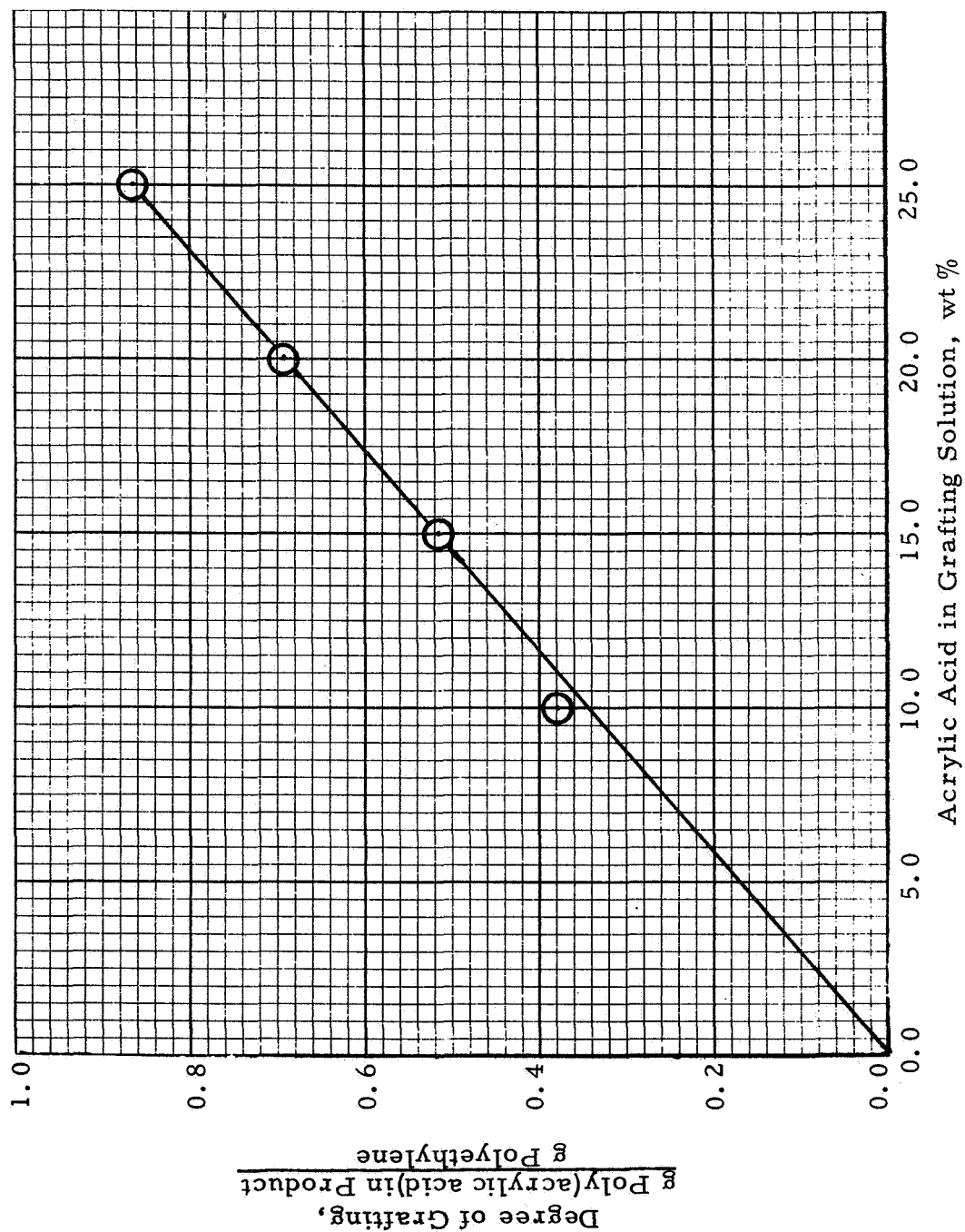


FIGURE 1. THE EFFECT OF ACRYLIC ACID CONCENTRATION IN THE GRAFTING SOLUTION ON THE DEGREE OF GRAFTING OF POLYETHYLENE FILM

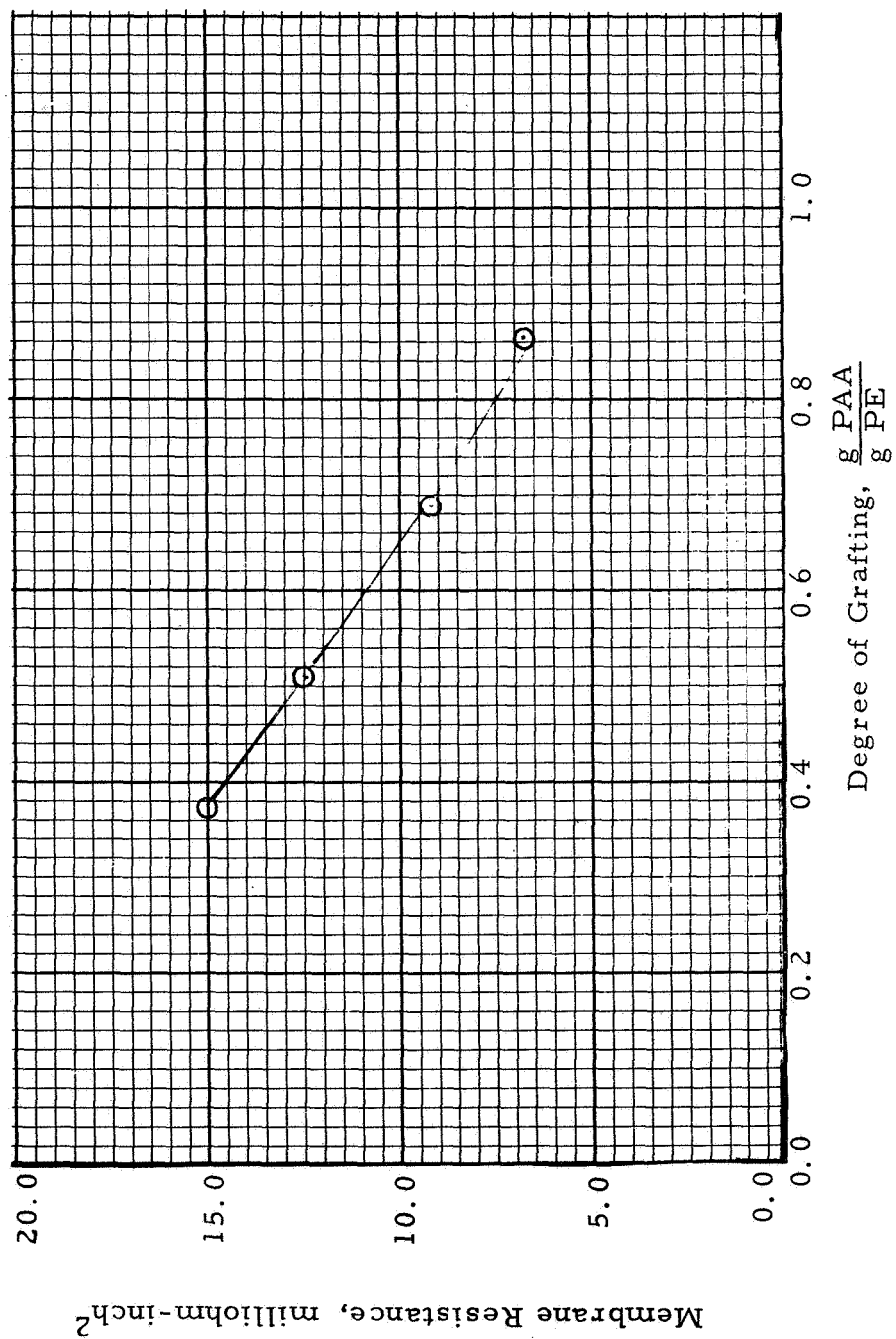


FIGURE 2. THE ELECTRICAL RESISTANCE OF ACRYLIC ACID-GRAFTED POLYETHYLENE IN 40% KOH AS A FUNCTION OF DEGREE OF GRAFTING