

Postirradiation polymerization of Vinyl Monomers on Poly(ethylene Terephthalate)

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Synopsis

A postirradiation process was evaluated in polymerizing vinyl monomers on poly(ethylene terephthalate) (PET) film, fiber, and fabric. The use of DMF, pyridine, and DMSO as swelling agents to facilitate monomer incorporation and effective polymerization were also investigated. The solvents were effective in promoting the incorporation of acrylic acid (AA) in PET film and that of *n*-vinyl-2-pyrrolidinone (VP) in Dacron 54. AA, a good swelling agent for PET, produced equivalent polymerization yield and moisture regain results with or without any solvent on Dacron 54 and 64. Polymerization yields on films increased with increasing total doses, but those on yarns and fabrics were independent of total dose. The different results obtained on film versus yarn and fabric from solvent treatment and total dose is thought to be due to the different surface-volume ratio of these substrates. The moisture properties of the substrates were dependent mainly upon the monomer type. Among the monomers studied, VP gave highest moisture regain values, followed by AA. The tensile properties of the modified Dacron 54 were not affected. However the breaking elongation of the modified Dacron 64 was slightly lowered by postirradiation polymerization without solvent treatment and was increased when solvent treatment was included.

INTRODUCTION

Poly(ethylene terephthalate) (PET) is one of the most radiation stable fiber-forming polymers. Upon gamma irradiation, two major forms of C[•] radicals produced have been suggested to be mostly on the paraffinic segment and to a much lesser extent on the aliphatic segment of the PET chains.¹⁻³ The aliphatic radicals of the polymer chain are formed directly because of the higher radiation sensitivity of the aliphatic segments than the aromatic. Aromatic radicals are subsequently produced from the collision with the aliphatic segments. The radicals on the paraffinic chains are formed mainly in the the crystalline regions, and the more stable radicals on the phenolic rings are formed in the amorphous regions. It is this latter form of radicals that predominates at low irradiation does up to 10 Mrad.⁴ Irradiation of PET in air has shown to produce chain scission at 0-1 and 12-500 Mrad.⁵ Crosslinking has been theorized⁶ and experimentally confirmed⁷ at the low dose range usually employed in gamma-irradiation polymerization. PET fiber has been found to lose few of its properties at low doses, and noticeable chemical degradation is not found at doses up to 20 Mrad.⁸

Polymerization of vinyl monomers in PET has been studied mostly using either the preirradiation or the mutual-irradiation processes. The former

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method involves preirradiating the substrates prior to the reaction with the monomer. The extent of grafting is normally low even with high radiation doses. In mutual irradiation, both polymer and monomer are present during irradiation. It is usually performed in aqueous solutions of monomers, and homopolymerization is controlled by adding appropriate inhibitors in the monomer solutions. The monomers evaluated with either process have included acrylic acid,^{9,10} acrylonitrile,^{9,10} hydroxyethyl methacrylate,⁹ methyl acrylate,⁹ methyl methacrylate,^{10,11} 2-methyl-5-vinyl pyridine,¹² vinyl alcohol,¹³ vinyl acetate,^{13,14} styrene,^{9,15,16} and several other vinyl monomers.¹² Low polymerization and difficulty of grafting have been contributed to the very low production of free radicals on the PET polymer chain.^{17,18}

In polymerizing a monomer in a polymer, the diffusion of the monomer into the polymer controls chain growth and chain termination in the internal matrix of the host polymer. To facilitate the incorporation of monomers into the host polymer, swelling agents can be used to open the physical structure of the host polymer.¹⁶ In a previous study, six organic solvents were used to promote the incorporation of acrylic acid in PET. Among the solvents studied, DMSO, DMF, and pyridine were found to be most effective in facilitating the incorporation of acrylic acid.¹⁹

PET fiber has very low chemical reactivity and water absorption properties due to its hydrophobic chemical structure and high crystallinity. In the present study, improvement of water absorption properties is attempted by polymerizing vinyl monomers in PET. A postirradiation process is employed in which the monomer is incorporated into the PET polymer matrix with the aid of swelling agents prior to the exposure of a gamma source. This process is similar to the pad-cure finishing process commonly used in the textile industry. The effects of swelling agent and radiation level on the extent of polymerization and the effects of monomer type on the moisture properties of the modified PET substrates are examined.

EXPERIMENTAL

The materials used in this study were experimental Mylar films (400 E from du Pont) and polyester fabrics (Dacron type 54 and type 64 from Test Fabrics, Inc.). Dacron type 54 is a disperse-dyeable homopolymer of PET, whereas Dacron type 64 is a disperse- and basic-dyeable copolymer of PET. The organic solvents used, *N,N*-dimethylformamide (DMF), dimethyl sulfoxide (DMSO), and pyridine (PY), were all reagent grade. Monomers studied were acrylic acid (AA), methyl acrylate (MA), vinyl acetate (VA), and *N*-vinyl-2-pyrrolidinone (VP). Acrylic acid was purified by being passed through activated neutral alumina. Sensitizers in MA and VA were removed by distillation. Inhibitor in VP was filtered with #50 hardened filter paper.

The fibrous substrates were rinsed with distilled water and dried in a vacuum oven at 60°C overnight. Films were degreased with trichlorotrifluoroethane (CCl₂FCClF₂). The initial dry weight W_i was obtained after drying in a vacuum oven at 105°C for 2 h. Solvent treatments were performed in a temperature-controlled silicone oil bath for a predetermined time. Excess solvent was removed by blotting the specimens between pieces

of filter paper to obtain the solvent incorporated weight W_s . The samples were then immersed in concentrated monomer solutions for predetermined time periods. A 10:1 (ml/g) monomer-to-sample ratio based upon the initial sample weight was employed. The samples were again blotted and weighed to obtain the monomer incorporated weight W_m . Each sample was immediately placed in a test tube, flushed with nitrogen for 30 min, and stoppered.

Polymerization was induced by a cobalt 60 gamma source. All polymerization work was conducted by triplicated specimens. Total irradiation doses up to 6.2 Mrad were achieved by varying the exposure time at a dose rate of 9.17 rad/s. Immediately after the irradiation, samples were first rinsed and then extracted with proper solvents for 4–6 h. Methanol was used to extract specimens polymerized with AA, MA, and VA, whereas water was used to extract those modified with VP. The polymerized weight W_p was measured after each sample was dried at 60°C overnight and at 105°C for 2 h in a vacuum oven. The polymerized samples were then conditioned at 21°C and 65% RH for 4 days to get the conditioned weight W_c .

The solvent uptake S , monomer uptake M , polymerization yield P , and moisture regain MR were calculated according to the following equations:

$$S\% = \frac{W_s - W_i}{W_i} \times 100\%$$

$$M\% = \frac{W_m - W_i}{W_i} \times 100\%$$

$$P\% = \frac{W_p - W_i}{W_i} \times 100\%$$

$$MR\% = \frac{W_c - W_p}{W_p} \times 100\%$$

Water contact angle measurements on films were performed with a microscope goniometer. The contact angles of 0.5 μ l doubly-distilled-deionized water droplets were taken between 30 and 60 s after the deposition of the droplets. The average of 10 measurements was used for each specimen. Tensile properties of the yarns were determined following ASTM 2256 procedures using an Instron. For each specimen, 15 yarn samples were used. In this study, all data reported are the means of measurements, with some standard deviations in parentheses. Surface morphology of the fibrous substrates was revealed by the use of a scanning electron microscope (SEM).

RESULTS AND DISCUSSION

Polymerization on Film as Affected by Solvent and Dose

Among the six solvents studied previously,¹⁹ PY was found to be most efficient and was followed by DMF and DMSO in facilitating the incorporation of AA in PET films. The influence of solvent and irradiation dose

on the polymerization of AA was thus evaluated with PY and DMF at doses ranging from 0 to 4.6 Mrad (Table I). Polymerization yield and polymerization efficiency were dependent upon the total dose in both solvent systems. The PY treatment produced much higher polymerization yield than DMF. These results were consistent with those obtained from plasma-induced polymerization of AA on the same PET films.¹⁹ The polymerization yields on the PY-treated films were much higher than the DMF-treated ones. The polymerization efficiency is somewhat higher for the DMF-treated films, however. This suggests that greater extent of polymerization product on the PY-treated films was actually removed by extraction. The higher polymerization yields on the films treated with PY mainly reflect the much greater monomer uptakes in these films.

Moisture regain indicates the hydrophilicity of the bulk of the substrate. Polymerization of AA with the DMF system apparently did not improve moisture regain or bulk hydrophilicity of the treated films. Pyridine, on the other hand, increased the moisture regain of the PE films several fold. In both solvent treatments, the moisture regain values are independent of the dose and polymerization yield. This seems to suggest that the improved bulk hydrophilicity are due to the effects of the solvent pyridine and its effect on increasing polymerization.

Surface water contact angles of these films were also measured and are shown in Figure 1. Untreated PET film has a water contact angle of 71.8° ($\pm 1.5^\circ$). The combination of solvent and monomer treatment without irradiation resulted in a considerable improvement on surface wettability as indicated by the 20° decrease in water contact angle for the treated surfaces. Again, surfaces of PY-treated films were more hydrophilic than those of DMF-treated ones. This observation suggests that surface hydrophilicity of films is predominately solvent-monomer related and is not as much affected by irradiation.

TABLE I
Effect of Solvents and Dose on Polymerization and Moisture Regain of Acrylic Acid Treated PET Films

	Solvent Uptake (%)	AA Uptake (%)	Dose (MRad)	Polymer yield (%)	Polymer efficiency ^a	MR (%)
Control	—	—	—	—	—	0.34 (0.11)
DMF ^b	9.7	10.8	0	1.1	0.09	0
	(0.2)	(1.3)	1.5	7.5	0.64	0.48 (0.21)
			3.1	8.6	0.97	0.43 (0.14)
Pyridine ^b	12.4	198.6	0	40.4	0.23	2.92 (0.24)
	(1.0)	(3.3)	1.5	95.4	0.39	2.10 (1.08)
			3.1	119.9	0.47	2.10 (1.53)
			4.6	143.8	0.72	2.75 (0.24)

^a Polymerization efficiency = % polymerization yield over % monomer uptake.

^b Solvent treatments were performed at 100°C for 30 min.

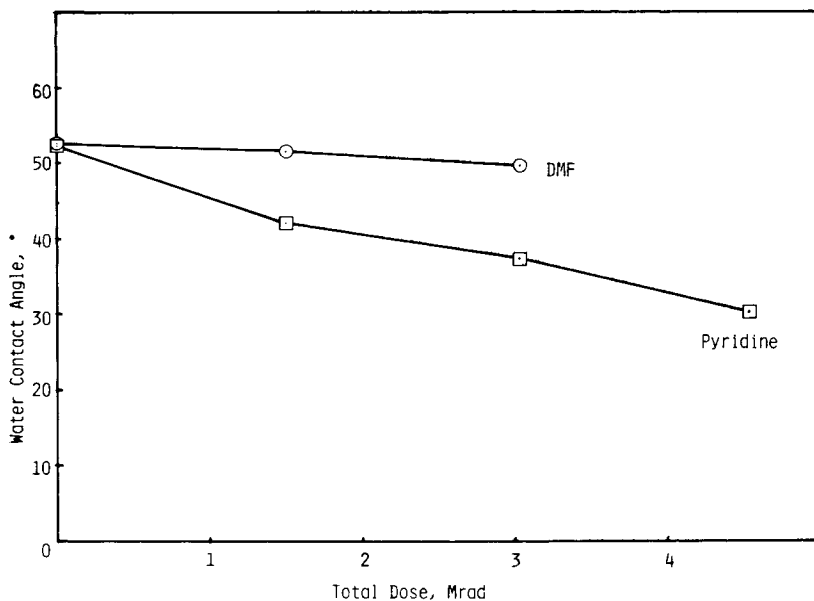


Fig. 1. Water contact angles (CA) of AA-polymerized PET films. Solvent treatments were performed at 100°C for 30 min. CA of the untreated PET films = 71.8°.

Polymerization on Yarn as Affected by Solvent, Monomer, and Dose

AA, VA, and MA were selected as monomers to be polymerized on Dacron type 64 yarns at doses up to 6.2 Mrad. A higher solvent uptake with DMF than with pyridine was found (Table II). The incorporation of each monomer did not vary between the two solvent systems. The above observation on the polyester yarns is different from that on the PET films. Dacron 64, a basic- and disperse-dyeable polyester, contains a small percentage of copolymers. The varying performance of the film and yarn during solvent and monomer treatments may have been due to the differences in their chemical constituents and substrate formation.

Acrylic acid uptakes were much higher than the other two monomers in either solvent system. This is probably because AA itself is a good swelling agent for PET. The subsequent polymerization yield and moisture regain data were much greater for acrylic acid than those for the other two monomers. A parabolic relationship between data on moisture regain and the polymerization yield was clearly demonstrated among AA, MA, and VA polymerized Dacron yarns (Fig. 2).

Similar to the results obtained from films, dose was found not to be a significant factor in influencing the degree of polymerization in yarns. The lowest dose studied, i.e., approximately 1.5 Mrad, was sufficiently energetic to induce a high degree of polymerization for acrylic acid on Dacron yarns. Since gamma irradiation can penetrate the bulk of thin film and fibrous substrates, the bulkiness of the materials should not interfere with the polymerization process. However, the polymerization on films was found dependent on dose, whereas that on yarns was independent of dose. This

TABLE II
Effects of Solvent,^a Monomer, and Irradiation Dose on the Polymerization
on Dacron Type 64 Yarns

Solvent	<i>S</i>	Monomer	<i>M</i>	Dose (Mrad)	<i>P</i>	<i>MR</i>		
DMF	38.8 (1.3)	AA	51.4 (4.5)	1.54	36.3	6.44		
				3.16	31.6	2.46		
				4.76	33.8	2.87		
				6.20	31.9	2.94		
				1.54	0.8	0.39		
				3.16	0	0.40		
		VA	7.5 (1.0)	4.76	-0.3	0.66		
						6.20	0.2	0.46
						1.54	2.3	0.38
						3.16	2.5	0.45
						4.76	1.6	0.37
						6.20	1.4	0.41
PY	23.7 (2.9)	AA	49.2 (2.6)	1.54	34.9	3.43		
				3.16	29.6	2.23		
				4.76	33.2	2.65		
				6.20	30.7	2.50		
				1.54	4.0	0.55		
				3.16	-0.9	0.36		
		VA	6.7 (0.4)	4.76	-0.4	0.34		
						6.20	0.2	0.53
						1.54	8.1	0.70
						3.16	3.1	0.31
						4.76	1.7	0.36
						6.20	1.5	0.49
MA	9.3 (3.0)	1.54	8.1	0.70				
				3.16	3.1	0.31		
				4.76	1.7	0.36		
				6.20	1.5	0.49		

^a Solvent treatment was performed at 100°C for 30 min.

may be explained by their difference in surface-volume ratios. Both chain propagation and termination reactions during polymerization could be more easily accomplished near the surface than in the bulk of the host polymer. A much greater surface-volume ratio on fibers facilitates not only the retention of monomer but also the polymerization of monomer, thus the higher extent of effective polymerization at the similar irradiation dosage.

Additional Solvent and Monomer Variables

Preliminary data on film and yarn have suggested the greater influence of solvent and acrylic acid over dose. Investigation was therefore focused on additional solvent and monomer variables. The effects of solvents conditions, that is solvent treatment duration and solvent temperature, on Dacron type 64 yarns were thus studied with AA polymerized at 1.50 Mrad. Table III shows the data obtained with various solvent conditions and with the monomer alone. Lengthening the solvent treatment time from 5 to 30 min at 100°C did not seem to cause major changes in the amount of solvent and monomer absorbed by the yarns, the polymerization yield, or the moisture regain data. The uptakes of solvent or monomer at a higher temperature were not different, either. However, negative polymerization yields

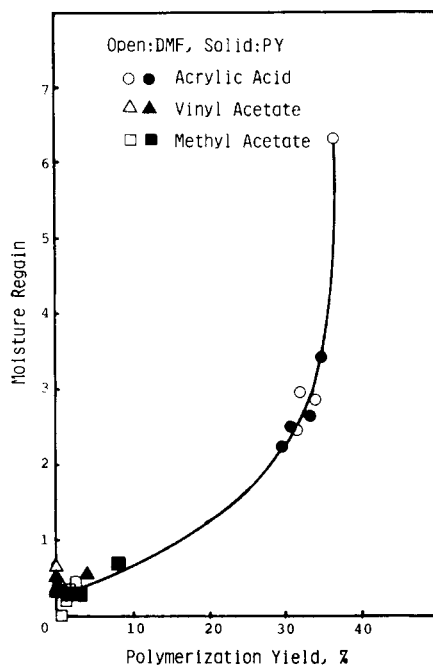


Fig. 2. Moisture regains of modified Dacron 64 yarns.

and low moisture regains were obtained at 140°C. This is probably due to extraction of low-molecular-weight PET and partial dissolution of PET in DMF at this higher temperature. These data also demonstrated that AA is not only a good swelling agent but also a monomer that can be polymerized effectively in polyester. DMF appeared to be a slightly better agent for polymerization on Dacron 64 than PY.

In a previous study,²⁰ the moisture regain of Nylon 6.6 was improved threefold using another vinyl monomer, *N*-vinyl-2-pyrrolidinone. DMSO, an

TABLE III
Effects of Solvent Treatment Time and Temperature on the Polymerization of AA on Dacron Type 64 Fabric^a

	Time (min)	S	M	P	MR
AA only	—	—	48.3 (8.3)	21.6 (4.0)	2.34 (0.17)
DMF, 100°C	5	40.1 (0.2)	46.4 (2.8)	25.1 (1.4)	3.03 (0.14)
	10	38.5 (0.9)	53.1 (4.5)	28.6 (2.8)	3.15 (0.04)
	30	37.7 (1.0)	47.7 (3.4)	22.7 (13.6)	3.67 (2.42)
Pyridine, 100°C	5	25.4 (5.4)	47.1 (2.2)	21.4 (1.9)	2.39 (0.15)
	10	27.5 (4.0)	46.6 (2.5)	19.1 (3.9)	2.27 (0.25)
	30	24.2 (1.0)	50.5 (1.5)	23.8 (11.9)	2.50 (0.92)
DMF, 140°C	2	40.0 (4.2)	34.2 (7.1)	-29.2 (5.6)	0
	5	41.8 (1.2)	43.4 (2.5)	-22.4 (0.5)	0.57 (0.47)
	10	46.3 (4.4)	45.4 (7.9)	-22.5 (2.9)	0.42 (1.13)

^aTotal dose = 1.50 Mrad. Moisture regain of the control yarns = 0.68 (0.04).

equally effective solvent as DMF for polymerization of AA on PET,¹⁵ was used for polymerizing VP on Dacron type 54 yarns and fabrics. Wider solvent temperatures at 60, 80, 100, 140, and 160°C were also evaluated. The trend among these temperatures was not clear for solvent uptake and monomer uptake in either yarn form or fabric form (Table IV). However, when the polymerization and moisture regain data were compared, it was apparent that treatment at 80°C gave the optimum results for yarns. For the fabrics, on the other hand, polymerization yield or moisture regain data did not seem to indicate any specific effect by varying thermal energy input. The somewhat higher and uniform polymerization among all solvent temperatures on fabrics may be because the newly polymerized polymer is in the form of coating on the fabric surface.

Polymerization of AA and VP on Fabric

The optimum postirradiation conditions determined on Dacron yarns were then employed to polymerize AA and VP on Dacron 54 and 64 fabrics. DMSO, the most effective in producing polyester with the highest moisture regain, was used. Table V lists polymerization data on both untreated and DMSO-treated polyesters. The effect of solvent is illustrated better when the solvent is used with a monomer that by itself is not a swelling agent for the host polymer, such as VP. DMSO appeared to be effective in promoting polymerization of VP on Dacron 54 but not on Dacron 64. The incorporation of a copolymer in Dacron 64 may have made it less responsive to DMSO than the homopolymer Dacron 54. On the other hand, when a monomer such as AA is swelling agent for the host polymer, the use of additional solvent (i.e., DMSO) does not affect the extent of polymerization. Overall, VP produced more hydrophilic materials than AA consistently with either fabric.

TABLE IV
Effects of DMSO Treatment^a Temperatures and Substrate Structures on the Polymerization of VP on Dacron Type 54

	Solvent Temperature (°C)	Dose (Mrad)	<i>S</i>	<i>M</i>	<i>P</i>	<i>MR</i>
Yarn	60	3.06	46.5 (1.4)	77.2 (16.5)	2.7 (0.6)	0
	80	3.46	50.9 (1.4)	83.6 (2.1)	37.0 (4.2)	5.21 (0.16)
	100	3.31	48.0 (0.7)	55.0 (1.8)	8.1 (1.2)	1.83 (0.21)
	140	3.31	58.0 (6.9)	71.9 (4.2)	14.4 (5.0)	1.32 (0.28)
	160	3.31	58.3 (1.5)	75.1 (5.4)	22.6 (3.8)	4.88 (0.91)
Fabric	60	3.46	36.2 (0.7)	64.3 (2.7)	30.8 (1.0)	6.04 (1.95)
	80	3.06	55.7 (2.1)	93.8 (10.9)	31.0 (4.8)	5.89 (0.52)
	100	3.46	51.6 (0.8)	62.7 (2.8)	27.9 (11.1)	4.50 (0.57)
	140	3.06	41.4 (3.0)	72.3 (2.2)	25.4 (4.7)	8.07 (0.73)
	160	3.06	34.6 (2.6)	51.4 (7.2)	23.0 (2.5)	5.62 (0.77)

^a Treatment time was 10 min.

TABLE V
Polymerization^a of AA and VP on Dacron Type 54 and 64 Fabrics

	Monomer	Solvent	S	M	P	MR
Dacron type 54	AA	None	—	80.7 (11.3)	56.2 (14.1)	2.7 (0.9)
		DMSO ^b	45.8 (1.9)	89.4 (7.9)	42.2 (5.7)	2.3 (0.5)
	VP	None	—	46.0 (8.0)	19.7 (4.1)	2.8 (1.5)
		DMSO	57.7 (4.3)	94.4 (17.3)	57.3 (5.7)	4.9 (0.8)
Dacron type 64	AA	None	—	51.3 (5.6)	28.3 (4.8)	2.9 (0.4)
		DMSO	45.6 (4.1)	78.0 (11.5)	35.1 (4.6)	1.3 (0.5)
	VP	None	—	63.9 (7.2)	40.6 (0.6)	3.8 (0.4)
		DMSO	45.6 (2.9)	63.3 (10.5)	40.3 (4.5)	3.6 (0.9)

^aTotal dose = 1.58 Mrad.

^bDMSO treatment at 80°C for 10 min.

Single-Fiber Tensile Properties of Modified Yarns

Table VI lists the single-fiber tensile properties of the polymerized Dacron yarns. For Dacron 64, the polymerization of both AA and VP without solvent pretreatment affected the tensile properties of the fiber similarly; that is both lowered the extension at break and the other properties remained unchanged. The penetration of monomer and the subsequent polymerization and/or crosslinking of monomers in the amorphous regions of the PET polymer may have contributed to the increase in rigidity of the host polymer matrix resulting in less extension at break.

On the other hand, polymerized Dacron 64 with solvent pretreatment in both PY and DMF had higher breaking elongation but other tensile properties remained consistent. DMF and PY, both strong reactive solvents for PET, may induce irreversible modification of the polymer morphology, such as void formation²¹ and higher crystallinity.^{22,23} Transformation of amorphous areas to crystalline areas can possibly reduce crosslinking sites on the host polymer chains. Greater freedom for chain movement is thus al-

TABLE VI
Single-Fiber Tensile Properties of Modified Dacron Yarns

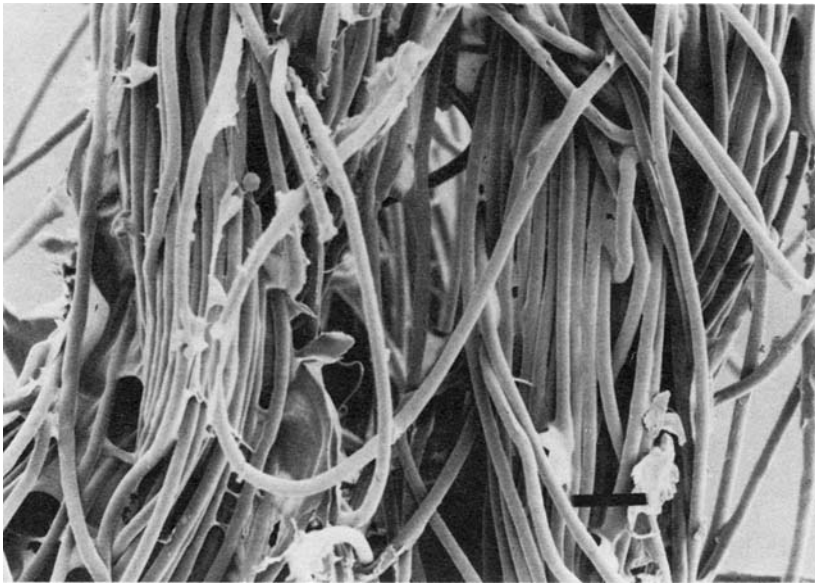
	Solvent	Monomer	Breaking load (g)	Breaking Elongation (%)	Yield load (g)	Yield Extension (%)
Dacron 64 ^a	None	None	14.9 (0.9)	27.1 (4.5)	3.2 (0.2)	2.1 (0.3)
	DMF ^b	AA	14.9 (0.7)	34.6 (2.1)	3.4 (0.3)	2.4 (0.1)
	<i>p</i> ^b	AA	14.5 (1.3)	34.5 (0.8)	3.7 (0.5)	2.5 (0.5)
	None	AA	14.2 (0.7)	21.7 (1.6)	3.7 (0.3)	2.0 (0.2)
	None	VP	14.1 (1.9)	21.7 (3.2)	3.2 (0.2)	1.9 (0.4)
	Dacron 54 ^c	None	None	6.3 (0.7)	27.4 (0.4)	0.9 (0.1)
DMSO ^d		AA	6.4 (0.8)	33.3 (16.3)	1.1 (0.1)	1.9 (0.6)
DMSO ^d		VP	6.8 (0.4)	34.5 (5.7)	1.2 (0.2)	1.8 (0.6)
None		VP	6.5 (0.4)	30.9 (7.7)	1.1 (0.2)	2.0 (0.7)

^aIrradiated at 1.50 Mrad.

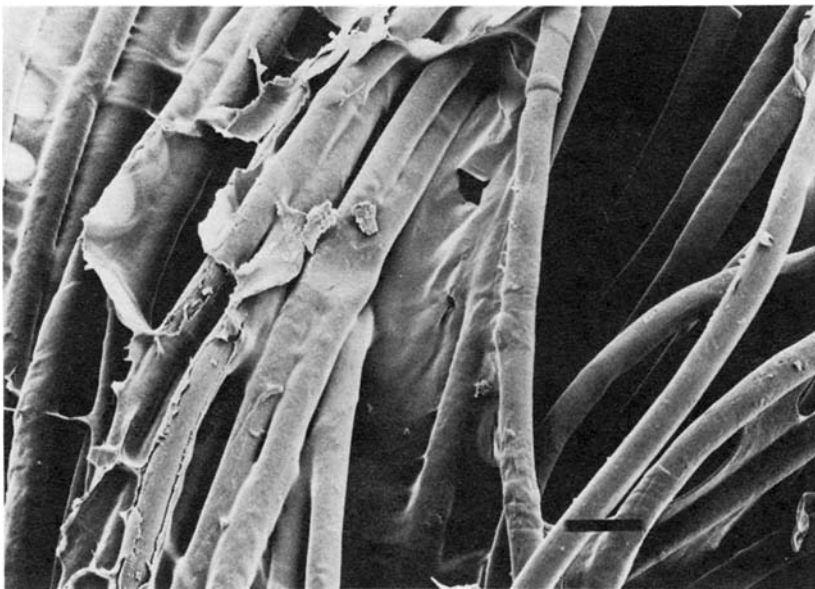
^bSolvent treatment at 100°C for 30 min.

^cIrradiation at 3.0 Mrad.

^dDMSO treatment at 80°C for 10 min.



(A)



(B)

Fig. 3. Surface morphology of modified yarns. (a) Dacron 54 polymerized with VP without solvent treatment at 3.0 Mrad: bar = 70 μm . (b) Dacron 64 polymerized with VP without solvent treatment at 1.5 Mrad: bar = 50 μm .

lowed for polymerization in the voids with minimum or no linkage to the host polymer chains. The plasticizing effect of the newly polymerized molecules in the amorphous region may also contribute to the higher elongation at break.

For the modified Dacron 54, however, no trend could be generalized on the tensile properties. None of the tensile properties was obviously affected. Greater standard deviation on breaking elongation data seems to be intrinsic to the irradiated Dacron 54 whether solvent and/or monomer were used or not.

Surface Morphology

Scanning electron micrographs of the modified yarns illustrate a considerable extent of coating on and bridging among fibers (Fig. 3). There appears to be more irregular formation on the modified fiber from the polymerization of VP. The polymerization of AA generally resulted in smoother surfaces with bridging among fibers.

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Received May 13, 1985

Accepted July 13, 1985