

# Flame-Retardant Brominated Styrene-Based Polymers. X. Dibromostyrene Grafted Latexes

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## INTRODUCTION

Natural and synthetic latexes are commercially used in a variety of coatings. In many coatings applications, the latexes with flame-retarding properties are desired. This applies in particular where latexes are used in textiles, carpetings, paints, clearcoatings, adhesives, sealants, caulks, nonwoven binders, etc.

Until recently, coatings users have generally had to use flame retardants that were developed for other applications. All of these products, either liquid or solid, have had an adverse effect upon the quality of the coating. The usual method by which flame-retardant (FR) properties are imparted to latexes is the blending-in of flame retardant additives, but disadvantages, such as phase separation upon aging, plasticization and migration to the surface, coagulation and sedimentation, generation of strong white pigmentation and settling out effect, toxicity resulting from the presence of antimony trioxide, have been associated with this approach. The best solution of these problems appears to be the elimination of additive flame retardants by incorporating flame retardancy into the polymer used for coating.

We have previously reported<sup>1-12</sup> the synthesis of a variety of dibromostyrene (DBS) -based FR latexes. These latexes were prepared from DBS and other monomers such as (meth)acrylic acids and their esters, butadiene, vinyl acetate, and styrene. Some of the challenges have been cost, flame retardancy, coating properties, latex stability, and monomer reactivity. However, if a practical and useful FR latex can be produced by simply grafting the DBS monomers onto a commercial latex or mixture of latexes, then the cost of producing such an FR latex can be significantly reduced.

Although a vast number of grafted polymer latex compositions has been described,<sup>13-29</sup> none of them have been identified with polymer latex modification with bromine for flame retardance. Some of the grafted latexes are claimed to have value in improving bacteria resistance for use in paints or other coating compounds,<sup>13</sup> improving adhesion of the modified latex to silicate glass, aluminum foil, cellophane, and textiles,<sup>20</sup> increasing the

*This paper describes the synthesis, characterization, and some applications of flame-retardant dibromostyrene grafted latexes. These latexes are synthesized by an emulsion polymerization technique. One of the prime factors to be considered in the choice of a commercial latex or a latex blend to be grafted is the glass transition temperature(s) of the polymer(s) in the final latex desired. The graft latexes were characterized in terms of glass transition temperature, solids content, bromine content, grafted dibromostyrene content, and flame retardancy. They are useful for a wide range of applications, including fabric backcoatings, carpetings, paints, adhesives, sealants, caulks, nonwoven binders, etc.*

viscosity of the latex and its activation energy of viscous flow,<sup>23</sup> improving impact strength,<sup>28,29</sup> and latexes being useful in paper coating compositions.<sup>27</sup>

This paper summarizes two U.S. patents<sup>30,31</sup> and two papers published in the *Polymer Preprints*.<sup>32,33</sup> It describes the synthesis, characterization, and application of DBS graft latexes. Latexes chosen for grafting are commercial latexes, such as Rhoplex HA-24 and HA-8, Hystretch V-

Table 1—Synthesis of DBS Homopolymer (PDBS) Latex

Description	Parts
Deionized (DI) water .....	180
SDS .....	3
Potassium persulfate .....	0.2
DBS .....	100
t-DDM .....	0-0.2

Seal under nitrogen in 8 oz. bottle. Rotate about a horizontal axis for 2-5 hr in water bath at 50°C. Remove from water bath. Cool to room temperature.

Presented at the 23rd Annual Waterborne, High-Solids, and Powder Coatings Symposium, on February 14-16, 1996, in New Orleans, LA. \*P.O. Box 2200, West Lafayette, IN 47906.

**Table 2—Synthesis of DBS Graft Commercial Latexes (Except for PB Latex)**

Description	Parts	Remarks
Commercial latex .....	84.5-45	Dry basis
Potassium lauroate .....	3	Blend
DBS .....	15.6-55	
CHP .....	0.2	
TEPA .....	0.4	
t-DDM .....	0.5	
DI water .....	14-57.3	Dilute to final solids of 40-65%

Seal under nitrogen in 8 oz bottle. Rotate about horizontal axis for 2-4 hr in water bath at 50°C. Remove from water bath. Cool at room temperature. The results of these preparations are set forth in Table 4.

29, Airflex 465, 4500, 4514 and 4530, Pliolite SBR latex and polybutadiene (PB) latex.

The graft latexes were characterized in terms of glass transition temperature ( $T_g$ ), solids content, bromine content, grafted dibromostyrene content and flame retardancy.

They are useful for a wide variety of applications, including fabric backcoatings, carpetings, paints, adhesives, sealants, caulks, non-woven binders, etc.

## EXPERIMENTAL

### Materials

Dibromostyrene, a product of the Great Lakes Chemical Corp., contains about 82% DBS, 12-13% monobromostyrene, and 5-6% tribromostyrene by weight. Ethyl acrylate (EA), acrylonitrile (AN), butyl acrylate (BA), styrene (S), acrylic acid (AA), and methacrylic acid (MAA) were used directly as received from Aldrich Chemical Co. Lauric acid, potassium hydroxide, sodium dodecyl sulfate (SDS), potassium persulfate, ammonium persulfate, sodium bisulfite, tetraethylenepentamine (TEPA), t-dodecyl mercaptan (t-DDM) were also used directly as received from Aldrich. Cumene hydroperoxide (CHP) from Elf Atochem and tetrahydrofuran (THF) from Baxter were used without further purification.

### Characterization

Table 4 lists composition and physical properties of DBS grafted commercial latexes. The DBS graft latex was characterized in terms of  $T_g$ , solids content, bromine content, and flame retardant property. The  $T_g$  was measured by DSC at a rate of 10°C/min. Percent bromine was measured by Schöniger Combustion Method (ASTM E 442-91). This test covers the determination of chlorine, bromine, or iodine in organic compounds by the oxygen flask technique. Flame retardant property was measured by MVSS-302 flammability test when used in polyester fabric backcoatings.

The MVSS-302 test criteria, a horizontal burn test, must be met by materials used in the interior of passenger cars. A specimen 14 in. x 1/2 in. is marked at a point 1-1/2 in. from each end. One end of the specimen is ignited in accordance with test procedures, and the

amount of time it burns measured from the time flame from the burning specimen reaches the first mark. Timing is stopped at the time flaming ceased or at the time at which the flame reaches the second mark. The test calls for a burn rate (measured as the flame processes from the first mark to the second) of less than four inches per minute. A rating of self-extinguishing (SE) requires no burn beyond the first mark (1-1/2 in.). A rating of rapid burn (RB) requires that the flame travels to the second mark or a burn length of 10.0 in. that is the distance between the first and the second marks.

Table 5 lists percent DBS grafted onto commercial latexes. Percent grafting of dibromostyrene onto commercial latexes, Pliolite SBR latex, and PB latex (Goodyear Tire & Rubber Co.) was determined by THF solvent extraction: the DBS-grafted latex, 55/45-DBS/SBR or 60/40-DBS/PB, was coagulated in a 1-3% MgSO<sub>4</sub> solution under stirring. The coagulated polymer was filtered, washed thoroughly with water, and dried overnight in a vacuum oven at 50°C to a constant weight. Two grams of the dried coagulated polymer was weighed in a cellulose extraction thimble (43 mm x 125 mm) and successively extracted with THF for 48 hr.

## FLAME RETARDANCE TESTING AND RESULTS

### Flame Retardancy of Physical Blend vs. Graft Latex

The PDBS latex was physically blended with an ungrafted commercial latex, Rhoplex HA-24 or HA-8 to result in a latex blend containing 30 and 25% DBS, respectively. Both latex blends, coated onto an 8 oz/yd<sup>2</sup>, 100% polyester fabric at a weight of 1.4-1.6 oz/yd<sup>2</sup>, failed the Motor Vehicle Safety Standard (MVSS) 302 flammability test [49 CFR Ch. V. (10-1-95 Edition)] (rapid burn (RB), worst rating). At a comparable DBS level, both DBS-grafted Rhoplex HA-24 and HA-8 passed the MVSS-302 flammability test at this add-on weight (self-extinguishing (SE), best rating).

### Textile Backcoating A

The 70/30-Rhopex HA-24/DBS graft latex was coated onto an 8 oz/yd<sup>2</sup> 100% polyester fabric at a weight of 1.6 oz/yd<sup>2</sup>, after oven drying at 100°C for 30 min. The coating was clear and slightly firmer than the commercial latex, which is considered satisfactory. The coated fabric passed MVSS-302 flammability test with an SE (best

**Table 3—Synthesis of DBS Graft PB Latex**

Description	Parts	Remarks
PB latex .....	40	Dry basis
SDS .....	1	
Potassium persulfate .....	0.2	
Sodium bisulfite .....	0.2	
DI water .....	20	
DBS .....	60	
t-DDM .....	0.5	

Seal under nitrogen in 8 oz bottle. Rotate about horizontal axis for 6.5 hr in water bath at 50°C. Remove from water bath. Cool to room temperature. The final latex has 57.9% solids (97.6% conversion), -79°C  $T_g$  and 34.34% Br (Table 4).

**Table 4—Composition and Physical Properties of Grafting of DBS onto Commercial Latexes**

Example	Commercial Latex <sup>a</sup>	$T_g$ , °C			DBS Charged <sup>b</sup> wt%	Solids %	Br %	MVSS-302 Rating <sup>c</sup>
		Comm. Latex Reported Value	Comm. Latex Observed	Graft Latex Observed				
1	Rhoplex HA-24	-7	-17.2	-16.7	30.0	40-65	17.17	SE
2	Rhoplex HA-8	-10	-17.3	-15.0	25.0	40-60	14.31	SE
3	Hystretch V-29	-29	-37.6	-35.3	40.0	40-50	22.89	SE
4	Airflex 465	-5±3	—	—	25.0	40-50	14.21	SE
5	Airflex 4500	3	—	—	20.0	40-50	11.36	SE
6	Airflex 4514	12	—	—	20.0	40-50	11.45	SE
7	Airflex 4530	29	—	—	15.6	40-50	8.93	SE
8	Pliolite SBR latex	-55	-56.0	-55.2	55.0	50-60	31.35	SE
9	Polybutadiene latex	-80	-79.5	-78.9	60.0	50-60	34.34	SE

(a) Rhoplex HA-24 & HA-8 from Rohm & Haas Co.; Hystretch V-29 from BFGoodrich Co.; Airflex 465, 4500, 4514, and 4530 from Air Products and Chemicals, Inc.; Pliolite SBR and polybutadiene latex from Goodyear Tire & Rubber Co.

(b) Weight % DBS based on 100% total solids.

(c) Motor Vehicle Safety Standard (MVSS) 302 flammability test; SE = self-extinguishing.

rating) and a burn distance of 0.8 in. The same fabric backcoated with 1.6 oz/yd<sup>2</sup> of the commercial latex (Rhoplex HA-24) failed the MVSS-302 test with a burn distance of 10.0 in. (RB).

### Textile Backcoating B

When 1.4 oz/yd<sup>2</sup> of 70/30-Rhoplex HA-8/DBS graft latex was coated onto the 8 oz/yd<sup>2</sup> fabric, an SE rating was obtained with a 1.4 in. burn length. The commercial latex without modification (Rhoplex HA-8) failed to pass the MVSS-302 test at this add-on weight and yielded a burn distance of 10.0 in. (RB).

### Textile Backcoating C

When coated onto an 8 oz/yd<sup>2</sup>, 100% polyester fabric, Hystretch V-29 latex at 3.7 oz/yd<sup>2</sup> yielded a failure in the MVSS-302 test (10.0 in. burned). The 60/10/30-Hystretch V-29/S/DBS graft latex produced a 1.6 in. burn length at 3.7 oz/yd<sup>2</sup>. The graft latex had good flexibility and yet was slightly firmer than the latex ungrafted.

### Contact Adhesive

The 80/20-Rhoplex HA-24/DBS graft latex was coated onto 0.5 mil Mylar polyester film. After drying 90 sec at 158°F, two pieces of film were pressed together and rolled to eliminate any bubbles. For comparison the same procedure was followed using the commercial latex (Rhoplex HA-24). Upon cooling, the films were pulled apart by hand. No significant difference in adhesion between two samples was noticed and the adhesion was judged to be adequate. Flammability was tested by wrapping the film composite loosely about a four-inch diameter cylinder of fiberglass batt insulation. A three-inch blue methane Bunsen burner flame was placed against the lower and side surfaces of the batt/Mylar construction which had been positioned at a 45°C angle from vertical. There was no observable after flame when the burner flame was removed after 10-15 sec of contact. A similar construction utilizing the commercially available latex emulsion without DBS grafted to it was also tested for flammability. When the burner flame was

removed after 10-15 sec, flaming of the substrate continued until extinguished about a minute later with water.

### Latex Binder

A non-woven polyester fiber filter medium weighing about 1.25 oz/yd<sup>2</sup> was immersed in the 84.4/15.6-Airflex 4530/DBS graft latex. Upon drying at 300°F for 90 sec, the weight of the bonded fiber increased to 2.4 oz/yd<sup>2</sup>. A second sample of non-woven polyester fiber filter was immersed in the commercially available latex (Airflex 4530) to which DBS had not been grafted. Upon drying, the bonded fiber weighed 2.3 oz/yd<sup>2</sup>.

Both bonded fiber samples were tested for flammability by exposure to a four-inch high, 1950°F propane flame from a Fisher burner. The filter media were held 2-1/2 in. over the top of the burner at a 15°C angle from horizontal. The sample which was not bound with latex to which DBS was grafted burned the entire 12-in. length. The sample containing DBS graft latex burned between 8 and 9 in. prior to self-extinguishing.

### Latex Sealant

Two latex sealants were prepared. The first was 95/3/2-EA/AN/AA latex prepared according to the procedure described in the Latex Sealant section of reference (33). The second was 80/20-the first latex/DBS graft latex prepared by the general procedure for synthesis of DBS graft commercial latexes.

To 100 parts of each of the two latexes mentioned previously were added 2 parts Triton X-405 (Union Carbide), 23 parts benzyl butyl phthalate, 4 parts Varsol #1 (Exxon), 2.5 parts ethylene glycol, 2 parts Composition T dispersant (Calgon), 120 parts calcium carbonate (2 micron average particle size), and 2.5 parts titanium dioxide. Both caulks showed good adhesion to substrates including wood, glass, and concrete by spot adhesion test (ASTM D 3808). In this test, a thin-bladed stainless steel spatula or probe is used to separate the caulk spot from the substrate.

The flammability of each was tested by placing a bead of caulk 1/4 in. in diameter on a 1/2 in. wide

**Table 5—Percent DBS Grafted onto Commercial Latexes**

Example	Charge Ratio DBS/Latex	Latex Coagulated			Extract			Unextracted			DBS Grafted <sup>a</sup> %
		wt, g	Br, %	PDBS, %	wt, g	Br, %	PDBS, g	wt, g	Br, %	DBS Grafted g	
10	SBR latex	2.004	—	—	0.895	—	—	1.164	—	—	—
11	55 PDBS/45 SBR latex blend	1.100 PDBS	—	—	1.383	43.62	1.062	0.673	<0.01	—	—
		+ 0.900 SBR	—	—							
12	55 DBS/45 SBR	2.000	31.67	55.74	0.726	36.75	0.469	1.273	29.23	0.655	59.5
13	PB latex	2.003	—	—	0.451	—	—	1.554	—	—	—
14	60 PDBS/40 PB latex blend	1.200 PDBS	—	—	1.447	39.90	1.016	0.624	<0.1	—	—
		+ 0.800 PB	—	—							
15	60 DBS/40 PB	2.000	34.34	60.44	1.117	34.35	0.707	0.893	22.85	0.359	29.9

(a) % DBS grafted =  $\frac{\text{wt. of DBS grafted (unto commercial latex)}}{\text{wt. of DBS charged}} \times 100$

For example 12:  $\frac{29.23}{56.82} \times 100 = 51.4\%$  — % of DBS grafted unto SBR latex in the unextracted portion.

$1.273 \text{ g} \times 51.4\% = 0.655 \text{ g}$  — wt. of DBS grafted

$\frac{0.655}{1.1} \times 100 = 59.5\%$  — % DBS grafted

1.1  
(DBS monomer used contains 56.82 wt.% bromine.)

strip of asbestos cement board. The caulk was subjected to the Butler chimney flammability test (ASTM D 3014). The first caulk, which did not contain DBS grafted to the latex, burned the full 250 mm of the test specimen. The caulk containing DBS burned less than 160 mm.

### Latex Paint

Two latex paints were prepared. The first was 30/68/2-S/BA/MAA latex prepared according to the procedure described in the Latex Paint section of reference (33). The second was 80/20—the first latex/DBS graft latex prepared by the general procedure for synthesis of DBS graft commercial latexes.

In an open vessel with continuous high shear mixing, 40 parts by weight water, 0.15 parts antifoaming agents, dispersants and surfactants Tamol 731 (0.4 parts), Triton X-207 (0.28 parts), and Silwet L-7602 (0.28 parts) (Tamol is a product of Rohm and Haas, Triton and Silwet are products of Union Carbide), 0.62 parts sodium polyacrylate thickener, 18 parts titanium dioxide, 28.3 parts calcium carbonate, and 7.34 parts 1-butanol were added. Mixing speed was then reduced, and 50 parts latex was added. An additional 4.6 parts water, 1.15 parts methyl cellosolve, and 3 parts sodium polyacrylate thickener were then added. Composition A contained the first latex without DBS grafted on; composition B contained latex with DBS.

Paint compositions A and B had good adhesion to the Mylar film and film forming properties (ASTM D 3730). This test measures the resistance of removal of the coating from the Mylar film when scuffed or scraped. Each was used to coat a 1 mil Mylar film to a 10 mil wet thickness. The dry film measured about 3 mils. The dry film/Mylar was tested by the limiting oxygen index test (ASTM D 2863). Composition A without DBS had a

limiting oxygen index of 22. Composition B with DBS had an oxygen index of 24, a significant improvement in flame retardancy.

### DISCUSSION

This paper demonstrates that commercial latexes can be grafted with DBS monomer to produce FR latex products useful in a wide range of coating applications. Criteria include the  $T_g$ , physical properties, applications, and cost. One of the prime factors to be considered in the choice of a latex or a latex mixture to be grafted is the  $T_g$ (s) of the polymer(s) in the final latex desired. Based on these criteria, we selected available commercial latexes, including acrylic latexes (Rhoplex HA-24 and HA-8, and Hystretch V-29 for binders, textile backcoatings, and adhesives), vinyl acetate-ethylene latex (Airflex 465 for adhesives), latexes of ethylene-vinyl chloride-third monomer which imparts amide functionality to the polymer (Airflex 4500, 4514, and 4530 for adhesives), SBR latex (Pliolite SBR for adhesive, foam, and rug), and butadiene rubber (BR) latex (PB latex for tires, retreads, and mechanical goods). We also synthesized acrylic latexes suitable for sealants and paints. Commercial latexes with  $T_g$ s greater than +29°C for modification were not chosen, because grafting of a high  $T_g$  (+141°C) PDBS onto commercial latexes will increase the  $T_g$ s of the final latexes. They will lose their original physical properties and become brittle, stiff, and not result in film formation upon coating.

As shown in Table 4, all the DBS-grafted commercial latexes (40-65% solids, 8.93 to 34.34% Br) passed the MVSS-302 flammability test with an SE rating. There is little difference between the observed  $T_g$ s of the commercial latexes and the observed  $T_g$ s of the DBS grafted latexes. We speculate that this could be attributed to the

short DBS grafted side chains. It is noteworthy that DBS grafted PB latex prepared by a redox system of potassium persulfate and sodium bisulfite passed the MVSS-302 flammability test (SE), whereas the graft latex prepared by a redox system of CHP and TEPA failed (RB, worst rating). This indicates that the former has a higher grafting efficiency than the latter as shown by the data in Table 5.

In order to understand the DBS grafting efficiency on the commercial latexes, we must select a solvent which dissolves ungrafted commercial polymers and homopolymerized DBS (PDBS) (ungrafted), but does not dissolve DBS grafted polymers. After the solvent extraction, the grafted portion is separated from the mixture of ungrafted portion. By analyzing the bromine contents in two separated portions in each sample, we can then calculate how much DBS has been grafted on the commercial latexes.

Two out of nine DBS-grafted commercial latexes, 55/45-DBS/Pliolite SBR (ex. 12, Table 5), and 60/40-DBS/PB latex (ex. 15, Table 5), were selected for THF solvent extraction to determine the percent DBS grafted (Table 5). The DBS homopolymer (PDBS) can be extracted by THF completely (ex. 11 and 14, Table 5). Thus, the unextracted PDBS residue could be considered to be grafted onto the commercial latex. The percent DBS grafted is given by the weight of grafted (or unextracted) PDBS divided by the weight of DBS charged. As shown in Table 5, ~ 60% DBS was grafted onto SBR and ~ 30% was grafted onto PB. These data may explain why DBS grafted SBR latex gave better flame retardant property than did DBS grafted PB latex.

Indeed, our results indicate that DBS can be readily grafted onto commercial latexes. However, % DBS grafted may depend on reaction conditions, initiators, and the composition of the latexes.

In our past experience,<sup>34</sup> FR efficiency of the DBS-based latexes is higher than that of a physical blend of FR DBS latex (PDBS) and a non-FR latex. In the present work, it was observed that FR efficiency of a DBS graft commercial latex is also higher than that of a physical blend. For example, at a comparable DBS content, both DBS grafted Rhoplex HA-24 and HA-8 latex coated 8 oz/yd<sup>2</sup> 100% polyester fabrics at a weight of 1.4-1.6 oz/yd<sup>2</sup>, passed the MVSS-302 flammability test with an SE (best rating) while a physical blend of ungrafted Rhoplex HA-24 or HA-8 latex with DBS homopolymers latex (PDBS) failed the test (RB, worst rating) (see Flame Retardance Testing and Results Section).

We plan to carry out research on solvent extraction of every DBS grafted latex other than DBS grafted SBR and BR latexes to determine DBS grafting efficiency. The structure of the various grafted polymers can be studied by electron microscopy, selective staining, cold stage, freeze fracture, and cryomicrotomy. Sometimes these techniques are not yet sufficiently developed to yield a conclusive picture of the polymer structure. Spectroscopy (NMR, FTIR, ESCA) and chromatography (GC, GPC, HPLC, SEC, TLC, IIP, CHDF) continue to evolve in characterizing the graft latexes.

## CONCLUSIONS

Dibromostyrene can be readily grafted onto nine commercial latexes plus two acrylic latexes via an emulsion polymerization technique. For example, THF solvent extraction of DBS grafted SBR and BR latexes yielded 60 and 30% DBS grafted, respectively. These data may explain why the former latex gave better flame retardancy than did the latter latex.

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All DBS grafted commercial latexes yield coatings with good flame retardancy without significant diminution of other physical properties. They are useful for a wide variety of applications in fabric backcoatings, contact adhesives, latex binders, latex sealants, and latex paints.

## ACKNOWLEDGMENTS

The authors are obliged to Jane Likens for the preparation of graft latexes, Xiaomin Zheng for the solvent extraction of the DBS-grafted latexes, and Rich S. Rose, David E. May, and David J. Honkomp for the coating formulation experiments and flammability testing.

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