Studies on the Polyblends of Poly(vinyl Chloride) with Various Methacrylate Copolymers, Physical and Mechanical Properties. I.

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Synopsis

The effect of blending various methacrylate copolymers on the physical and mechanical properties of poly(vinyl chloride) (PVC) has been investigated. Copolymers of methylmethacrylate with methylacrylate, ethyl acrylate, butyl acrylate, and 2-ethylhexyl acrylate in 80:20 and 50:50 wrt methylmethacrylate have been prepared and characterized by nuclear magnetic resonance spectroscopy. Polyblends of PVC and such polyacrylates have been prepared in 80:20 ratio by melt blending technique and characterized by thermomechanical analysis to study the glass transition behavior vis-à-vis the compatibility of these blends. Mechanical properties of these blends revealed a substantial increase in impact strength particularly when long chain acrylate polymers like butyl acrylate and 2-ethyl hexyl acrylates are used; however, there is a decrease in the yield stress and initial modulus. A shift from brittle failure to ductility has been observed in blends of PVC on incorporation of these acrylate copolymers. Scanning electron microscopic studies have been carried out to support these observations.

INTRODUCTION

The inherent problems of poor impact strength and difficult processing of rigid PVC has been reported to be overcome by the process of blending with certain other polymeric materials which are referred to as impact modifiers and processing aids.¹ These processing aids when added to PVC result in substantial improvement in processing without significant detraction from other properties. Several types of polymers function as effective processing aids. Among them the most important types are polymers composed of acrylates and methacrylates. Adding a small quantity of these materials causes only minor changes in physical properties of PVC. However, incorporation of a greater quantity of certain acrylic copolymers acts both as processing aids as well as impact modifiers.² The incorporation of acrylic copolymers in PVC is analogous to the rubber toughening of glassy polymers. Impact strength has been reported to have increased by mechanically mixing under appropriate conditions of such polymers with PVC. Various acrylate polymers like poly(ethyl acrylate), poly(butyl acrylate), and poly(2-ethyl hexyl acrylate)

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are well known to be rubbery in character having glass transition temperatures T_g close to or below room temperature. On the other hand, there are amorphous, rigid, and glassy polymers like poly(methyl methacrylate) and poly(ethyl methacrylate) which possess very high values of T_g and are sometimes used as processing aids in PVC. The low T_g acrylates have some difficulty of incorporation in PVC due to limited compatibility and they adhere too strongly to metallic surfaces.³ The high T_g acrylates obviously cannot improve the toughness and impact properties. However, the use of copolymers made from these two categories for blending with PVC offer the possibility of improving processing behavior as well as toughness without loss in rigidity and strength. An example of this can be a copolymer of methyl methacrylate and butyl acrylate or 2-ethyl hexyl acrylate. The low T_g acrylate fraction of the copolymer would be expected to increase impact strength while poly(methyl methacrylate) would help in maintaining the rigidity and tensile strength in PVC-based blends.

The improved properties of such blends have been suggested by various workers due to the specific interactions like hydrogen bonding and dipole–dipole interactions between molecules of the constituent polymers.⁴ Blaga and Feldman,⁴ Barlow and Paul,^{5,6} and others^{7,8} have suggested that the α -hydrogen of vinyl chloride can interact with the ester carboxyl group (H-bond acceptor of the acrylic terpolymer) to form a hydrogen bond and is also a key factor in achieving miscibility. The close value of the solubility parameters of PVC and PMMA and various acrylate copolymers also account for the compatibility of blends. The solubility parameters (δ) those given in Table I have been calculated using the equation $\delta = \rho \epsilon Fi/M$, where values of Fi have been taken according to Hoy's method.⁹

The present study aims on the preparation of polyblends of poly(vinyl chloride) and copolymers of methyl methacrylate with methyl acrylate, ethyl acrylate, butyl acrylate, and 2-ethyl hexyl acrylate followed by their characterization. The thermal, mechanical, and the scanning electron microscope studies of these blends have also been carried out.

Sample	$\frac{\delta}{(cal/cc^{1/2})}$
Poly(vinyl chloride)	9.47
Poly(methylmethacrylate)	9.27
Poly(methyl acrylate)	9.82
Poly(ethyl acrylate)	9.22
Poly(butyl acrylate)	9.16
Poly(2-ethyl-hexyl acrylate)	8.20
Poly(MMA-co-MA) 80:20	9.38
Poly(MMA-co-MA) 50:50	9.54
Poly(MMA-co-EA) 80:20	9.26
Poly(MMA-co-EA) 50:50	9.24
Poly(MMA-co-BA) 80:20	9.24
Poly(MMA-co-BA) 50:50	9.21
Poly(MMA-co-EHA) 80:20	9.02
Poly(MMA-co-EHA) 50:50	8.73

 TABLE I

 Calculated Solubility Parameters of PVC and Acrylate Polymers

EXPERIMENTAL

PVC resin as supplied by M/s NOCIL Ltd. Bombay, India, having mol wt (M_v) 65,000 was used for the present studies.

Methyl methacrylate (MMA), methyl acrylate (MA), ethyl acrylate (EA), butyl acrylate (BA), and 2-ethyl hexyl (EHA) acrylate (BDH, LR Grade) were first washed with 5% NaOH to remove inhibitor and then with distilled water to eliminate traces of alkali. The monomers were then distilled under reduced pressure and dried over anhydrous sodium sulfate before use.

Benzoyl peroxide (BDH) was purified by dissolving in chloroform at room temperature and crystallized at low temperature by adding methanol.

Monosodium and disodium phosphate (BDH), acrylamide (Superfloc A 100, American Cynamide), chloroform (BDH AR), and octyl tin mercaptide (M/S ALA Chemicals) were used as received.

Synthesis of Methacrylate Copolymers

Suspension polymerization technique was followed to prepare the copolymers of MMA with MA, EA, BA, and EHA. Two sets of experiments have been carried out where comonomers are in 80:20 and 50:50 ratio with respect to MMA and the above-mentioned acrylates.

Polymerization reactions were carried out in a round bottom flask of 1-L capacity. The reaction was followed in aqueous medium in which polyacrylamide was used as suspending agent and mono- and disodium phosphate as buffers. An inert atmosphere was maintained throughout the reaction. Meanwhile the temperature of the contents was raised to the required level and weighed quantities of MMA and other acrylates along with benzoyl peroxide (initiator) and dodecyl mercaptan (chain transfer agent) were poured in sequences. Reaction was continued for 3 h at 78°C. The copolymers in each case obtained were filtered, washed with hot water, and finally dried over vacuum at 50°C for 24 h.

Preparation of Polyblends

PVC/poly(MMA-co-acrylates) blends in 80:20 ratio were prepared by melt blending technique. The mixture of polymeric materials along with stabilizer (2 phr) and lubricant (1 phr) for PVC, viz., octyl tin merceptide and stearic acid, respectively, were mixed on a two-roll mill at 150° C for 10 min. Subsequently the blends were compression-molded on a hydraulic press at 170° C using 300 kg/cm² pressure. Molded sheets of about 3 mm thickness were cooled under ambient conditions. Dumbbell-shaped specimens were prepared for tensile testing (ASTM D 638) while notched samples were prepared for impact strength (ASTM D 256).

Tests and Measurements

Nuclear magnetic resonance (NMR) spectroscopy of methacrylate copolymers was carried out on a Perkin-Elmer NMR, 90 MHz, using CDCl_3 as the solvent at room temperature.

Glass transition temperature measurements were made of PVC/P (MMAco-acrylate) blends on thermomechanical analyzer (TMA), Stanton Redcroft Model 691, by heating sample from a temperature of -150 to 220° C at a heating rate of 10° C/min. The low temperatures were attained by using liquid air as cooling medium.

Impact testing was done on an Izod impact tester at room temperature i.e., $25 \pm 1^{\circ}$ C. A minimum of five samples with a notched angle 45° C were tested in each case.

Tensile properties were measured on an Instron Universal Tester (Model 1121) at ambient temperature using gage length 5 cm, crosshead speed 5 mm/min, and chart speed 20 cm/min. A minimum of five samples were tested in each case.

Scanning electron micrographs of impact-fractured surfaces were recorded on a steroscan Model S4-10 of Cambridge Scientific Instruments Ltd.

Characterization of Methacrylate Copolymers

Characterization of methacrylate polymers in two different ratios, i.e., 80:20 and 50:50, with respect to MMA was done by nuclear magnetic resonance spectroscopy and thermomechanical analysis.

The copolymers synthesized are expected to be random in nature and the composition of the copolymer is approximately equal to the composition of monomer feed. Reactivity ratios for copolymerization of MMA with acrylate esters are such as they would give ideal random copolymers (MA, $r_1 = 0.93$, $r_2 = 0.99$), (EA, $r_1 = 0.47$, $r_2 = 1.83$) and $r_1 = 0.37$, $r_2 = 2.8$).¹⁰ The values suggest as $r_1 \approx r_2 = 1$ so that the selectivity exhibited by a polymer radical for either monomer would result in a random copolymer. All the copolymers had an intrinsic viscosity η_0 in the range of 0.24 dL g⁻¹ to 0.28 dL g⁻¹ in chloroform at 25°C. Exact values of M_v could not be found because of nonavailability of K and α values for methacrylate copolymers. PMMA has $\eta_0 = 0.24$ dL g⁻¹ and consequently $M_v = 85,000$, as calculated by equation:

$$\eta_0 = k M^{lpha}$$

Nuclear Magnetic Resonance (NMR) Studies

Figure 1 presents the NMR spectrum of PMMA. In case of PMMA three peaks which appear at highest field represent the methacrylate methyl groups of varying tacticity. Thus a band at 0.90 ppm indicates syndiotactic methyl groups, the band at 1.06 ppm arises from atactic methyl groups, and the weak band at 1.25 ppm represents the resonance of isotactic methyl groups. The corresponding isotactic, atactic, and syndiotactic methylene groups absorb in chemical shifts ranging from 1.4 to 2.4 ppm. The methyl ester group is represented by the sharp peak near 3.65 ppm which is present in all the spectra by virtue of the presence of MMA. NMR data of PMMA is in good agreement with published data.¹¹

In case of P(MMA-co-MA) (Fig. 2) the band at 0.8-0.90 ppm represents the syndiotactic methyl groups and hand at 1.4-2.4 ppm indicates methene group of methyl ester. In P(MMA-co-EA) (Fig. 3) the ethyl groups display their characteristic high field triplet at 1.26 ppm and low field quartlet pattern at 4.12 ppm. In P(MMA-co-BA) (Fig. 4) weak triplet at 4.03 ppm represents the center peak of the butyl terminal methyl group. The acrylate methylene



Fig. 1. NMR spectrum of poly(methylmethacrylate) at room temperature.

group absorb in the band range of 1.4–2.4 ppm. Two internal methylene group overlap with the butyl absorbance bands in the range from 1.1 to 1.9 ppm. The band at 0.92 ppm probably represents the distorted triplet of terminal $-CH_3$ of the butyl group. In P(MMA-co-EHA) (Fig. 5) a cluster of peaks from 3.8 to 4.1 ppm represent, the hexyl terminal methyl group. The three methylene groups of hexyl side chain are in range of 1.1–1.9 ppm. The band at 0.92 ppm represents the distorted triplet methyl of the hexyl group.



Fig. 2. NMR spectra of poly(MMA-co-MA): (A) 80:20 copolymer; (B) 50:50 copolymer; at room temperature.



Fig. 3. NMR spectra of poly(MMA-co-EA): (A) 80:20 copolymer; (B) 50:50 copolymer; at room temperature.



Fig. 4. NMR spectra of poly(MMA-co-BA): (A) 80:20 copolymer; (B) 50:50 copolymer; at room temperature.



Fig. 5. NMR spectra of poly(MMA-co-EHA): (A) 80:20 copolymer; (B) 50:50 copolymer; at room temperature.

From various NMR spectra it is revealed that the characteristic peaks of PMMA in general and methyl ester peak in particular (which appears at 3.6 ppm) are present in all the copolymers. However, the characteristic peak in the case of copolymers, viz., the ethyl group in P(MMA-co-EA), the butyl terminal group in P(MMA-co-BA), and methylene groups of hexyl side chain and distorted triplet of methyl of hexyl group in P(MMA-co-EHA) are present.

Thermal Analysis

A comparison of glass transition temperature values of various acrylate polymers reflects that T_g goes on decreasing as the acrylates change from lower to higher alkyl groups. Such a transition can be explained on the basis of the structures of these polymers.¹⁰ With the increase in the ester group from MA to EHA, there is more segmental motion of the polymer chains at room temperature imparting elastomeric nature to the resin which is characterized by T_g well below the room temperature. It is also the amount of low T_g acrylate in the copolymer which effects the T_g .

 T_g of the acrylate copolymers has been calculated by the equation¹²

$$1/T_{g(\text{copolymer})} = W_1/T_{g1} + W_2/T_g2$$

 W_1 and W_2 are the weight fraction of two monomer in the copolymer and T_{g1} and T_{g2} are the respective glass transition temperature of each homopolymer.

Sample			T_g of PVC Blends (°C)					
	T_g of Copolymers (°C), calculated		PVC/80:20 polyacrylate copolymer		PVC/50:50 polyacrylate copolymer			
	80:20	50:50	Calculated	Experimental	Calculated	Experimental		
P(MMA-co-MA)	81.0	50.6	80.2	78	73.5	70		
P(MMA-co-EA)	69.0	29.0	77.7	78	68.5	62		
P(MMA-co-BA)	57.0	4.0	75.0	76.0	61.0	60		
P(MMA-co-EHA)	54.3	-1.0	74.4	68.0	60.17	58		

TABLE II Glass Transition Temperature (T_{e}) (°C) of PVC/Poly(MMA-co-Acrylate) Blends^a

^a PVC 75°C; PMMA 100°C.

RESULTS

Blends of PVC and Methacrylate Polymers: Thermomechanical Analysis

Thermomechanical analysis was carried out to study the effect of methacrylate copolymers on PVC. It is evident that incorporation of such copolymers in PVC as impact modifier is expected to impart significant plasticizing effect reducing its T_g . T_g of the blends has been calculated as is done with random copolymers using eq. (1). A single T_g has been observed in all the blends and both the calculated and experimental values are reported in Table II. From TMA a single endothermic transition that corresponds to the T_g of PVC and PVC/polymethacrylate blends is shown in Figures 6 and 7. Beyond T_g , the polymer blends are in the rubbery state till they melt and finally decompose at a temperature around 220°C.

Tensile Properties

Rigid PVC at ambient temperature is a stiff material and it breaks in a brittle manner, although it fails by a necking type of rupture. A look at the stress-strain properties of PVC/polyacrylate blends (Figs. 8 and 9) reveals that incorporation of the methacrylate copolymer in the PVC matrix results in decrease in tensile strength and tensile modulus but in a significant increase in elongation at break.

In general, in stress-strain curves there is a linear increase of stress with strain with gradual change of slope up to the position of the yield peak. Beyond the tensile yield peak there is a necking region followed by long drawing or immediate break depending on the nature of the blend. Area under stress-strain curve reveals a constant increase as chain length of the acrylate copolymer is increased. The role of an acrylate modifier in a glassy polymer like PVC is significant in shifting the behavior of a brittle material to an elastic or rubberlike material. The values of tensile strength elongation at break, tensile modulus, and hardness, and so on, are shown in Table III. From stress-strain data, it can be seen that yield stress is highest in case of PVC with least elongation at break, but it goes on gradually decreasing as one of the constituents of the acrylate copolymer (which is blended with PVC) is



Fig. 6. TMA thermograms of PVC and PVC/P(MMA-co-acrylate) blends (80:20 copolymer).



Fig. 7. TMA thermograms of PVC and PVC/P(MMA-co-acrylate) blends (50:50 copolymer).



Fig. 8. Stress-strain properties of PVC and PVC/P(MMA-co-acrylate) blends (80:20 copolymer), at room temperature.

changed from MA to EHA. If we observe the values of yield stress, which is a true stress at load observed at a comparatively low elongation of the sample, there is a regular yield region in stress-strain curves. The slope of the curve changes as the chain length of the acrylate copolymer constituent increases. Hence there is a change in the elastic modulus of the material which also gives a fair idea about the reduced rigidity. The area under the yield peak is a measure of the energy absorbed in the process of yielding. The energy can be correlated with the apparent toughness of the material and thus has a relationship with impact strength. This is called "work of yield." The effect of various acrylate copolymer on the work of yield or toughness has been shown in Figure 10. As can be seen from yield peak, there is not much difference between PVC and poly(MMA-co-MA) blended material, although there is a slight increase in the initial modulus which can be attributed to the glassy and



Fig. 9. Stress-strain properties of PVC and PVC/P(MMA-co-acrylate) blends (50:50 copolymer), at room temperature.

Sample	Impact strength (kg/cm/cm)	Maximum yield stress (kg/cm ²)	Yield strain (%)	Tensile modulus (kg/cm ² × 10 ³	Area under stress strain curve (arbitrary units)	Hardness Shore D
PVC	2.2	564	4.5	17.2	66	80
80:20 copolymer						
PVC/P(MMA-co-MA)	2.75	551	5.0	21.75	78	85
PVC/P(MMA-co-EA)	3.75	508	5.0	15.10	97	80
PVC/P(MMA-co-BA)	4.25	463	5.0	10.95	127	80
PVC/P(MMA-co-EHA)	11.25	310	4.5	8.85	176	75
50 : 50 copolymer						
PVC/P(MMA-co-MA)	5.0	466	5.0	18.20	110	80
PVC/P(MMA-co-EA)	6.0	400	5.0	11.42	123	75
PVC/P(MMA-co-BA)	13.5	277	4.0	8.59	178	75
PVC/P(MMA-co-EHA)) 39.25	260	4.0	8.20	740	70

TABLE III Mechanical Properties of PVC/Poly(MMA-co-Acrylate) Blends

rigid nature of poly(methyl methacrylate), which forms a major part of the copolymer. However, as we change from MA to EA, there is a considerable decrease in the yield peak and tensile modulus. In fact, this behavior is identical to that of rubber toughened glassy polymers. Incorporation of BA and EHA marks a significant change in the stress-strain behavior. While there is a great decrease in the yield stress and initial modulus, the material has got a wide yield peak and, after yielding, the material elongates considerably just like a rubber solid, although it cannot be presumed exactly to be the drawing phenomenon as observed in rubbers.

It is suggested that during the yielding process a part of the energy is absorbed by the dispersed polyacrylate fraction of the blend, which is rubbery in nature and thus undergoes sufficient elongation before fracture. The quantity of the rubbery acrylate polymer present is a decisive factor in determining the nature of the fracture. While PVC and PVC blends with lower acrylates show brittle fracture, the blends where long chain acrylates are incorporated, ductility is introduced in the system and the fracture is ductile. Further, the fracture behavior of these blends can be studied on the basis of scanning electron microscopy.

Impact Strength

The impact strength of PVC is considerably increased on blending with various methacrylate copolymers. The results are shown in Figure 11. The impact strength increases as the blending components change from MA to EHA. In case of blends containing the copolymers in 80:20 ratio with respect to MMA, the increase is not significant while in case of 50:50 copolymers there is a considerable increase in impact strength. The increase in the impact strength can be attributed to the low T_g of the acrylate copolymers within the PVC matrix.

In case of PVC and P(MMA-co-MA) blend, an insignificant increase in the impact strength may be due to the fact that PMMA, which is a major



Fig. 10. Plot showing 'work of yield' vs. number of carbon atoms in the side chain of acrylate copolymer constituent in PVC/P(MMA-co-acrylate) blends, at room temperature.



Fig. 11. Izod Impact strength of PVC and PVC/P(MMA-co-acrylate) blends: (A) 80:20 copolymer; (B) 50:50 copolymer; at room temperature.

component of the copolymer, is a stiff and brittle polymer and hence is not able to enhance the toughness in the final blend.

In case of PVC and P(MMA-co-EA) blend and PVC and P(MMA-co-BA), while there is a slight increase in the impact strength where 80:20 copolymer was used, an appreciable increase is observed in 50:50 acrylate copolymer blend. This is attributed to the introduction of flexibility in the acrylate copolymer by ethyl acrylate and butyl acrylate.

The outstanding increase in the strength is observed with a long chain acrylate like 2-ethyl hexyl acrylate. As can be seen from Figure 11, there is about a five times increase in Izod impact strength when the 80:20 copolymer is used and the increase is about 20 times that of PVC when the 50:50 copolymer is used.

Scanning Electron Microscopy (SEM) Studies

SEM micrographs of impact fractured surfaces of PVC and PVC/P(MMAco-acrylate) blends are shown in Figures 12 and 13. A brittle fracture is observed in the case of PVC with the surface showing microcharacteristics typical of a rigid and glassy surface. The methacrylate copolymers present are identified as fibrils, and large domains in the PVC matrix and a change in the surface morphology has been observed. The introduction of long fibrils and the coarseness of surface has been related to the ductility of the material.¹³ In the case of PVC/P(MMA-co-MA), there is not much difference in the fracture behavior with PVC, although some coarseness is introduced when the 50:50 copolymer is used in the final blend. In the case of PVC/P(MMA-co-EA), the fibrillar pattern is more conspicuous. The presence of a large domain of acrylate copolymer is visible in PVC/P(MMA-co-EA), in which the surface is



Fig. 12. Scanning electron micrographs of impact fractured specimens of PVC and PVC/P(MMA-co-acrylate) blends (with 80:20 copolymer).



(b)



Fig. 12. (Continued from the previous page.)

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(d)



Fig. 12. (Continued from the previous page.)



(a)



(b)

Fig. 13. Scanning electron micrographs of impact fractured specimens of PVC and PVC/P(MMA-co-acrylate) blends (with 50:50 copolymer).



(c)



Fig. 13. (Continued from the previous page.)

still more coarse. Domain size increases significantly in case of PVC/P(MMA-co-EHA) where surface is extremely ductile. In such a blend, as has been observed, the impact strength is maximum.

DISCUSSION

Incorporation of various methacrylate polymers in rigid PVC matrix have been shown to impart plasticizing effect in the otherwise glassy polymer, which is a key factor in achieving toughening in the final blend. Thermal analysis of PVC/polymethacrylate blends provides good information about the compatibility of the blends. The observation of a single T_g in PVC/polymethacrylate blends clearly shows that the two polymers are compatible giving a single phase when blended. However, glass transition behavior is not the only consideration for assuming a polymer blend to be completely compatible.

From the mechanical properties like tensile strength and impact strength, it can be argued that the role of an acrylate modifier in a glassy polymer like PVC is significant in shifting the behavior of a brittle material to an elastic or rubberlike material. The slope of stress-strain curve changes as the chain length of the acrylate copolymer constituent increases, suggesting a change in the elastic modulus of the material which gives a fair idea about the reduced rigidity. Increase in the initial modulus in case of P(MMA-co-MA) can be attributed to the glassy and rigid nature of the PMMA which forms the major part of the copolymer. However, in the case of other blends the behavior is identical to that of rubber-toughened glassy polymer.

In the case of rubber toughening of glassy polymers the microcrazing phenomenon is considered as the principal mechanism for increase in impact strength. In the present case, stress whitening and microphase separation is introduced as the chain length of the acrylate modifier increases. This also results in the decrease of the optical clarity of the blends. While PVC matrix remains transparent when lower acrylates copolymers are blended with it, the transparency is lost when more flexible acrylates like polybutyl and polyethyl hexyl acrylates are used. This can also be explained on the basis of solubility parameter calculations. As the absolute difference between the solubility parameters of PVC and acrylate copolymers increases, the transparency decreases (see Table I).

It seems probable why only a small amount of long chain acrylic ester is able to result in manifold increase in the impact strength of PVC. It was reported by Hardt¹⁴ Fleischer and co-workers,¹⁵ and later Haaf and coworkers¹⁶ that for optimum performance of a toughened PVC, the primary particles should be thoroughly dispersed in the rigid matrix. These modifier particles occupy the interstitial space between the chains of the rigid matrix, and the longer chain length of acrylate ester, the greater space they will occupy and consequently contribute more to absorb impact energy. The high concentration of modifier in this space may explain why in some cases only 5-10% of modifier may be effective.

CONCLUSIONS

1. PVC blends with copolymers of MMA and MA, EA, BA and EHA are compatible as shown by a single glass transition temperature (T_g) with improved impact strength.

2. Copolymers of MMA with MA and EA marginally increase the impact strength of PVC while copolymers of MMA and BA and EHA increase it significantly.

3. Copolymers of MMA-co-BA and MMA-co-EHA diminish the optical clarity of PVC due to less compatibility.

4. Ductility is introduced in PVC blends on incorporation of P(MMA-co-BA) and P(MMA-co-EHA).

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