

Investigation on Thermoplastic Co-poly(ether-ester) Elastomer Toughened Poly(methylmethacrylate) Blends

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ABSTRACT: Blends of poly(methyl methacrylate) (PMMA) and copoly(ether-ester) (COPE) elastomer have been prepared in different compositions namely, 95/5, 90/10, 85/15, and 80/20 wt % (PMMA/COPE), by melt mixing technique using twin screw extruder. The influence of COPE content on the mechanical properties especially impact strength, thermal behavior, and chemical resistance of PMMA have been investigated. The impact strength of the PMMA/COPE blends for all the compositions were found to be improved remarkably as compared to the virgin PMMA without affecting the other mechanical proper-

ties significantly. Various composite models, such as series model, parallel model, Halpin-Tsai equation, and Kerner's model have been used to fit the experimental mechanical properties. The effect of chemical and thermal ageing on the performance of the PMMA/COPE blends was also studied. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 109: 3511–3518, 2008

Key words: poly (methylmethacrylate); thermoplastic copoly (ether-ester); elastomer; toughened blends; impact strength; morphological behavior; chemical ageing

INTRODUCTION

Polymer blending is a useful technique for designing materials with a variety of properties. Blending of two or more polymers to create a material with properties beyond those achievable with a single polymer has become a challenging area of polymer science,¹ and it has become a popular route to develop materials with new properties.^{2–6} Polymeric blends consists of glassy matrix with dispersed rubber phase are known to exhibit improved impact properties.⁷ Toughness can be introduced either by incorporation of elastomeric phase during the polymerization or dispersion of elastomeric phase during compounding. It is well known that the impact strength of glassy polystyrene (PS) improves with the incorporation of elastomer such as polybutadiene rubber.^{7–10} Elastomer toughened plastics can be used to produce structural parts, which needs high impact strength and crack resistance for many engineering applications.

Among amorphous thermoplastics, poly(methyl methacrylate)(PMMA) is one of the most important polymers, distinguished by its versatile characteristics such as optical properties, hardness, high moduli, relative ease of processing, and rigidity with

good weathering resistance. Its limitations are brittleness and poor chemical resistance that needs to be improved to extend its range of applications. Such improvements are made by blending it with different types of elastomers for various use.

PMMA is a very well-known thermoplastic and many publications deal with the toughness of this brittle polymer.^{11–13} The effect of the rubber particle size on the plasticity of reinforced PMMA blends has been studied by Gloaguen et al.¹⁴ They considered PMMA blends containing latex particles with a soft core and a hard shell. Love11 et al. prepared multiple layer toughening particles as impact modifiers for PMMA.¹⁵ Franck and Lehmann made an important contribution to the understanding of rubber toughening of PMMA blends at different deformation speeds.¹⁶ Kilwon et al. have investigated the effect of interfacial adhesion on toughening behavior of rubber modified PMMA.¹⁷ PMMA have been toughened successfully by incorporating rubber particles.¹⁸ They achieved superior toughness values with the average particle size of 50 nm in 70:30 PMMA/rubber blend. Blends of PMMA with polyethylene terephthalate (PET) was studied by Kamata et al.,¹⁹ and their results shows good processability and high impact strength. Processability studies on thermoplastic elastomer (TPE) prepared by radiation-induced grafting of methyl methacrylate onto radiation vulcanized natural latex has been evaluated using kneader and hot roll mill at 150°C.²⁰ Schmidt et al. have studied the effect of composition

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of PMMA blends with styrene-butadiene-styrene (SBS) and PET.²¹ Bishara and Shaban have studied nonisothermal crystallization kinetics of PET and PMMA blends.²² It is also noticed that the modification of PMMA was carried out by *in situ* polymerization,²³ rubber modification,²⁴ and polyisoprene.²⁵ Fracture toughness and mechanical behavior of thermoplastic copoly(ether-ester) (COPE) elastomer toughened ABS was also been reported.²⁶ Recently, Poomalai and Siddaramaiah had reported on the improvement in impact strength and other properties of PMMA/TPU and PMMA/ethylene vinyl acetate (EVA) blends.^{27,28} Reports on PMMA blends with TPEs are scanty and there is an ample scope in the modification of PMMA.

Thermoplastic COPE has many of the most desirable characteristics of high-performance elastomers and flexible plastics. Its features are exceptional toughness and resilience; high resistance to creep, impact and flex fatigue; flexibility at low temperatures; high elasticity, and retention of properties at elevated temperatures. In addition, it resists deterioration from many industrial chemicals, oils, and solvents.

The objectives of this work is to develop COPE toughened PMMA by melt blending process to improve the impact strength and chemical resistance of PMMA without sacrificing other mechanical properties significantly. The obtained PMMA/COPE blends have been characterized for physicochemical, thermal properties, morphological behavior, and chemical resistance. Also the experimental values of tensile modulus and tensile strength were correlated with the theoretical (predicted) values derived from various proposed models.

EXPERIMENTAL

Materials

Poly(methyl methacrylate) (Gujpol-P 876-G) with 6 g/10 min melt flow index (MFI) and 1.19 g/cc density was procured from M/s Gujarat State Fertilizers Company Limited, India. Thermoplastic copoly (ether-ester) elastomer (Hytrel-4069) with 8.5 g/10 min MFI and 1.11 g/cc density was obtained from M/s Du Pont, USA.

Blend preparation

Initially the virgin polymers were predried in hot-air oven at 80°C for 4 h separately and dry blended with different compositions of PMMA/COPE before processing. The premixed PMMA with COPE with different compositions were melt mixed in microprocessor controlled 17.5 mm diameter twin screw corotating extruder (*L/D* ratio: 18:1) with granulation set-up

(Haake Rheocord 9000, Germany). The prepared PMMA/COPE blend compositions such as 100/0, 95/05, 90/10, 85/15, and 80/20 wt % were designated as HY0, HY5, HY10, HY15, and HY20, respectively. The processing temperature used was in the temperature range of 145–210°C and at 30 rpm screw speed for the blending operation. The resulting hot extrudate strands were quenched in a stream of cold water and pelletized. The granules were hot-air dried at 80°C for 4 h and made into test specimens as per ASTM standard specifications by injection molding process using computerized 50 ton injection molding machine (500-270 D model), Arburg, Germany. The molding temperature range and injection pressure used were 200–245°C and 100 bar, respectively.

Measurements

Tensile test was carried out on LR 100K model Universal Testing Machine (Lloyd, U.K.) as per ASTM D 638. Injection molded dumb bell-shaped specimens (165 mm × 13 mm) were used with cross-head speed of 5 mm/min. Notched izod impact strength test was performed by using izod-charpy digital Impact tester (model no.6546/000, Ceast, Italy) as per ASTM D 256. The specimen dimensions of 63.5 mm × 12.7 mm with notch radius of 0.25 mm were used for impact test. The physical properties such as density and hardness of the blends were measured by displacement method (ASTM D 792) and shore D hardness test method (ASTM 2240), respectively. The morphological behavior for the PMMA, COPE and their blends (HY5, HY10, HY20) were recorded using optical microscope (model No. SMC 76078) made by Setwa Optical, Tokyo, Japan. The melt flow index (MFI) test was carried out by using Advanced Melt Flow-Rate system (Rosand, UK) at 230°C and 1.2 kg load as per ASTM D 1238. Vicat softening temperature (VST) was measured by using VST apparatus (Wallace, England) as per ASTM D 1525. The chemical resistance test was conducted (ASTM D 543) by exposing the injection molded tensile specimens into the selected chemical reagents such as HCl, H₂SO₄, acetic acid, NH₄OH, Na₂CO₃, NaOH, *n*-hexane, and water for 168 h. The heat ageing was also done by using hot air oven as per ASTM-D 794 at 80°C for 168 h. The effect of chemical and heat ageing on tensile behavior of the blends have been evaluated and the results were correlated with unaged blends.

RESULTS AND DISCUSSION

Physicochemical properties

The measured properties such as density, MFI, impact strength, hardness, vicat softening temperature, and tensile behaviors of PMMA, COPE and

their blends are given in Table I. The variation of experimental and theoretical density (calculated) values as a function of blend composition is shown in Figure 1. The theoretical densities were calculated by volume additive principle, using the expression, $1/\delta = W_1/\delta_1 + W_2/\delta_2$; where δ is the density of the blend, δ_1 and δ_2 are the densities of the virgin components, and W_1 and W_2 are the weight fractions of the respective parent polymers. The density of blends decreased linearly with increase in COPE content. Recently, similar observations were made by Poomalai et al. for PMMA/EVA blends.²⁸ The experimental densities of PMMA/COPE blends are very close to the theoretical values and the variation is linear in nature. This may be due to the fact that the less polar COPE is incompatible with polar PMMA. The reduction in density may be due to the incompatibility, micro void formation, poor interfacial adhesion or phase separation between PMMA and COPE. However, the extent of reduction in densities marginally varies among the blends.

The MFI value increases from 1.35 to 1.72 g/10 min. as the COPE concentrations increases in the blends upto 10 wt % (Table I). This may be due to the reduction in softening temperature of the PMMA with the presence of elastomeric COPE phase which can act as a plasticizing agent. However, further addition of COPE in PMMA shows no further increase in MFI which may be due to the increase in melt viscosity at higher concentration of elastomeric phase in the blend. From Table I, it may be noticed that the gradual reduction in surface hardness with increase of COPE concentration in blends. This may be due to the presence of soft COPE component of very low hardness in the blend. The VST values of PMMA/COPE blends are lies in the range 95–96°C. This data indicates that there is no significant influence of blend compositions on VST data. A slight reduction in VST values of blends after incorporation of COPE was noticed. This can be attributed to the presence of flexible COPE component in PMMA matrix.

Impact strength

Impact strength evaluation is an important tool to study the toughening characteristics of rubber toughened plastics. The izod impact strength (notched) of PMMA and all its blends are given in Table I. The notched impact strength obtained for the PMMA is 19.1 J/m. The incorporation of COPE elastomeric phase into PMMA matrix leads to drastic improvement in impact strength of the blends from 19.1 to 42.89 J/m. The impact strength for all the blends are increased in comparison with PMMA, and at 10 wt % (HY10) addition of COPE shows remarkable improvement in impact strength (42.89 J/m) of about 125%. However, above 10 wt % addition of COPE

TABLE I
Physicomechanical Properties of PMMA, COPE, and PMMA/COPE Blends

COPE content in blends (wt %)	Blends designation	Density (kg/m ³) (±1.2%)	Tensile strength (MPa) (±4%)	Tensile modulus (MPa) (±5.5%)	Tensile elongation (%) (±5%)	Impact strength (J/m) (±5%)	Surface hardness (Shore-D) (±2%)	MFI (g/10 min)	Vicat softening point (°C)
0	HY0	1183	60.9	1280	10.96	19.1	90	1.35	96.5
5	HY5	1177	51.9	1077	37.31	26.0	81	1.50	96.0
10	HY10	1173	47.7	960	44.22	42.9	78	1.72	95.5
15	HY 15	1169	44.0	895	41.79	35.1	76	1.70	95.5
20	HY20	1166	37.8	755	35.95	34.5	72	1.61	95.0
100	HY100	1102	17.3	22	66.00	—	33	—	—

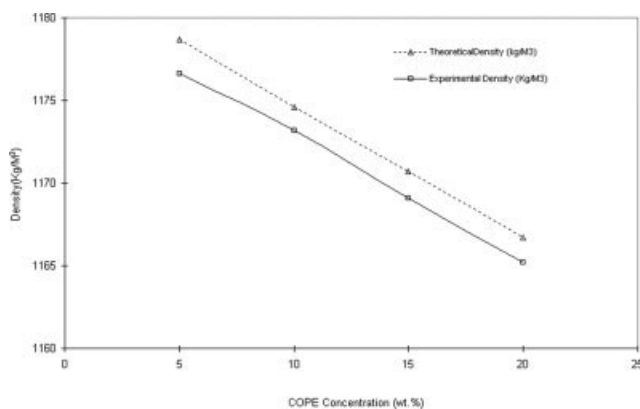


Figure 1 The plot of density versus COPE compositions of PMMA/COPE blends.

shows decrease in impact strength upto 15 wt % and then levels off by further addition of COPE. At higher concentration of rubber content changes the dispersion pattern to cocontinuous phase with the matrix which may leads to decrease in impact strength. The addition of 10 wt % COPE may be the optimum dispersion and adhesion in PMMA. It is generally been seen that the optimum dispersion and adhesion of the rubbery phase with plastics are the basic requirements of impact strength modifications.^{29,30} The word optimum signified sufficient compatibility i.e., neither total miscibility nor complete immiscibility between the components. The chemical nature of the dispersed phase and its compositions decides the dispersibility and adhesion with the continuous phase.

Tensile properties

From the tensile data (Table I) of the blends, it was found that the reduction in tensile strength and tensile modulus increase in the COPE concentration. However, the loss is not very significant upto 10 wt % of COPE content (HY10). The elongation at break increases remarkably in all the PMMA/COPE blends in comparison with PMMA. The reduction in tensile strength and modulus may be due to the plasticization effect caused by the addition of elastomeric phase. The extent of reduction in tensile strength and modulus are depending on the nature and concentration of rubbery phase.

Figure 2 shows the effect of COPE (wt %) content on tensile strength and surface hardness of PMMA/COPE blends. It is observed that the properties such as tensile strength and hardness lie more or less on the additivity line. However, it may be noted that a slight negative deviation upto 10 wt % of COPE concentration, which may be due to less interfacial adhesion between the constituents of the blends. However from 10 wt % of COPE content, the additivity line merges with the experimental results line, which

indicates better interaction between the PMMA and COPE materials. However, the reduction in tensile strength and modulus values are more or less follow linearity with the concentration of rubber content.

Many theories have been put forward for predicting the tensile modulus of heterogeneous blends.³¹ Various composite models, such as parallel model, series model, Halpin–Tsai equation, and Kerner’s model have been used to evaluate the mechanical properties of the blends. The highest upper-bound parallel model is given by the rules of mixtures:

$$M = M_1\varnothing_1 + M_2\varnothing_2 \quad (1)$$

where M is any mechanical property of the composite; M_1 and M_2 are the mechanical properties of components 1 and 2, respectively; and \varnothing_1 and \varnothing_2 are the volume fractions of components 1 and 2, respectively. This equation is applicable where the components are arranged parallel to one another so that an applied stress elongates each component by the same amount. The lowest lower-bound series model is found in the systems where the components are arranged in series with the applied stress. The mathematical expression for series model is:

$$1/M = \varnothing_1/M_1 + \varnothing_2/M_2 \quad (2)$$

According to the Halpin–Tsai equation,

$$M_1/M = (1 + A_i B_i \varnothing_2)/(1 - B_i \varnothing_2) \quad (3)$$

where,

$$B_i = (M_1/M_2 - 1)/(M_1/M_2 + A_i) \quad (4)$$

The subscripts 1 and 2 refer to the continuous phase and dispersed phase, respectively. The constant A_i is defined by the morphology of the system, for elastomeric domains dispersed in a continuous

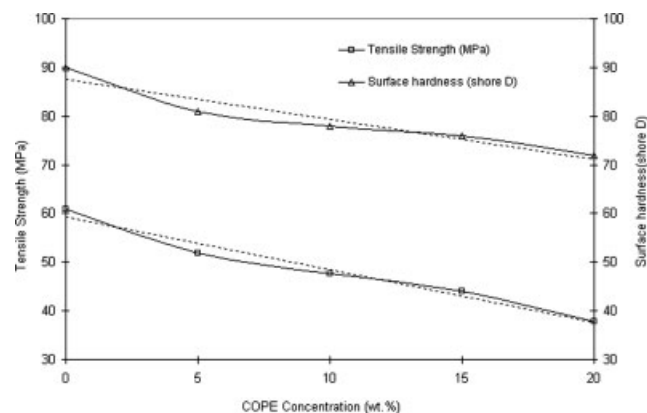


Figure 2 Effect of COPE compositions on tensile strength and surface hardness of PMMA/COPE blends.

matrix, $A_i = 0.66$. For an incompatible blend, mechanical properties are generally between the parallel model upper bound (M_U) and the series model lower bound (M_L).³² According to Kerner's Model,³³ when two polymeric systems have the same poisson's ratio ν and perfect adhesion at the boundary, the tensile modulus of blend is given by:

$$E_b = E_m \times \frac{\varnothing_d E_d / [(7 - 5\nu_m) + (8 - 10\nu_m)E_d] + \varnothing_m / 15(1 - \nu_m)}{\varnothing_d E_m / [(7 - 5\nu_m) + (8 - 10\nu_m)E_d] + \varnothing_m / 15(1 - \nu_m)} \quad (5)$$

The poisson's ratio of PMMA and COPE are 0.4 and 0.45, respectively. These values are close enough to use the Kerner equation in the above form for PMMA/COPE blends. The terms in eq. (5) have the standard notations.³³ E is the tensile modulus, \varnothing is the volume fraction of the discrete phase, and ν is the poisson's ratio. The subscripts b , m , and d refer to blend, the matrix, and the dispersed phase, respectively. All these models were used to estimate the tensile properties of PMMA/COPE blends theoretically. Figures 3 and 4 shows the experimental and theoretical curves of tensile strength and tensile modulus as a function of volume fraction of soft phase (COPE). The experimental values for tensile strength is very close to series model as shown in Figure 3. In Figure 4, the tensile modulus as a function of volume fraction of COPE phase shows that the experimental data are very close to Kerner's model.

Surface morphology

The morphology of heterogeneous polymer blends depends on blend compositions, viscosity of individual components, and processing history.³⁴ The blends with same processing history, the morphology are determined by the melt viscosity ratio and composition. Generally, the least viscous component

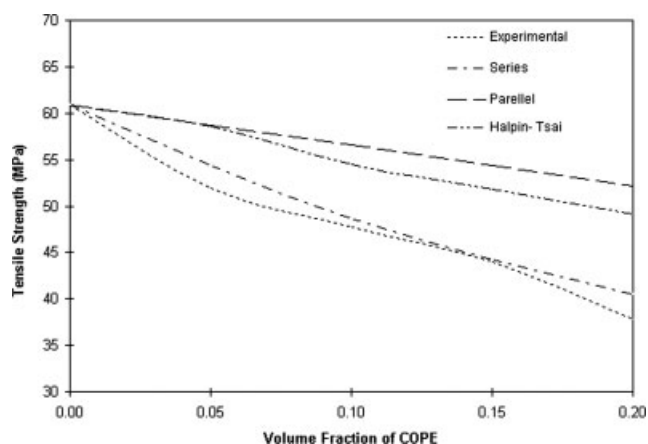


Figure 3 Experimental and theoretical values of tensile strength as a function of volume fraction of COPE phase.

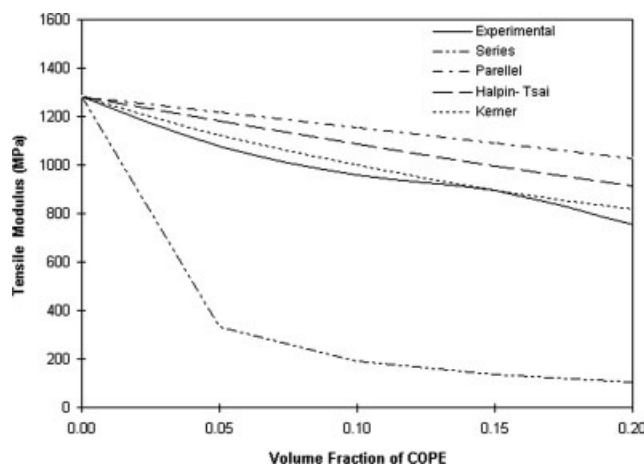


Figure 4 Experimental and theoretical values of tensile modulus as a function of volume fraction of COPE phase.

was observed to form the continuous phase over a larger composition range.³⁵

The optical micrographs of PMMA and COPE are given in Figure 5(a,b), respectively. These figures show two phase morphology for PMMA as well as

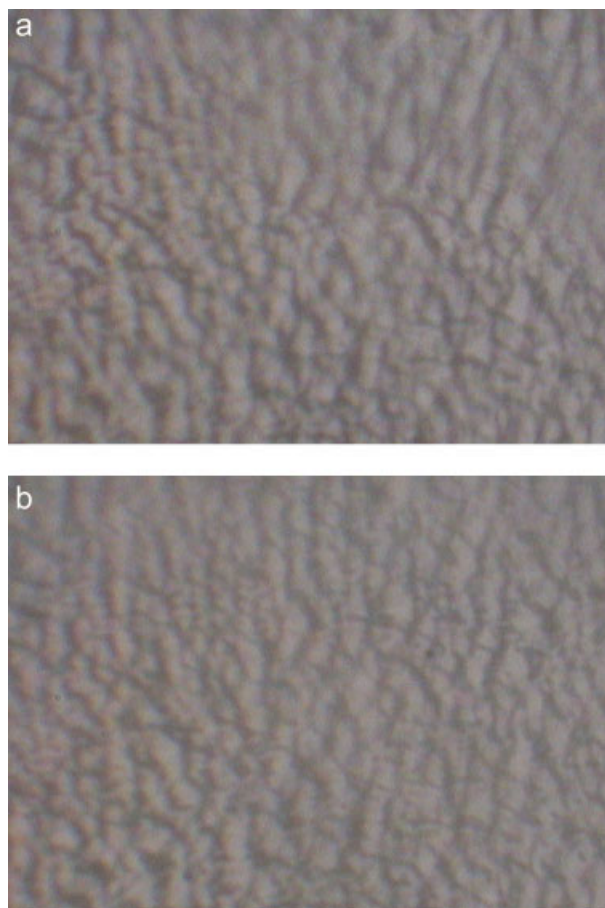


Figure 5 Optical micrographs ($\times 100$) of (a) PMMA and (b) COPE. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

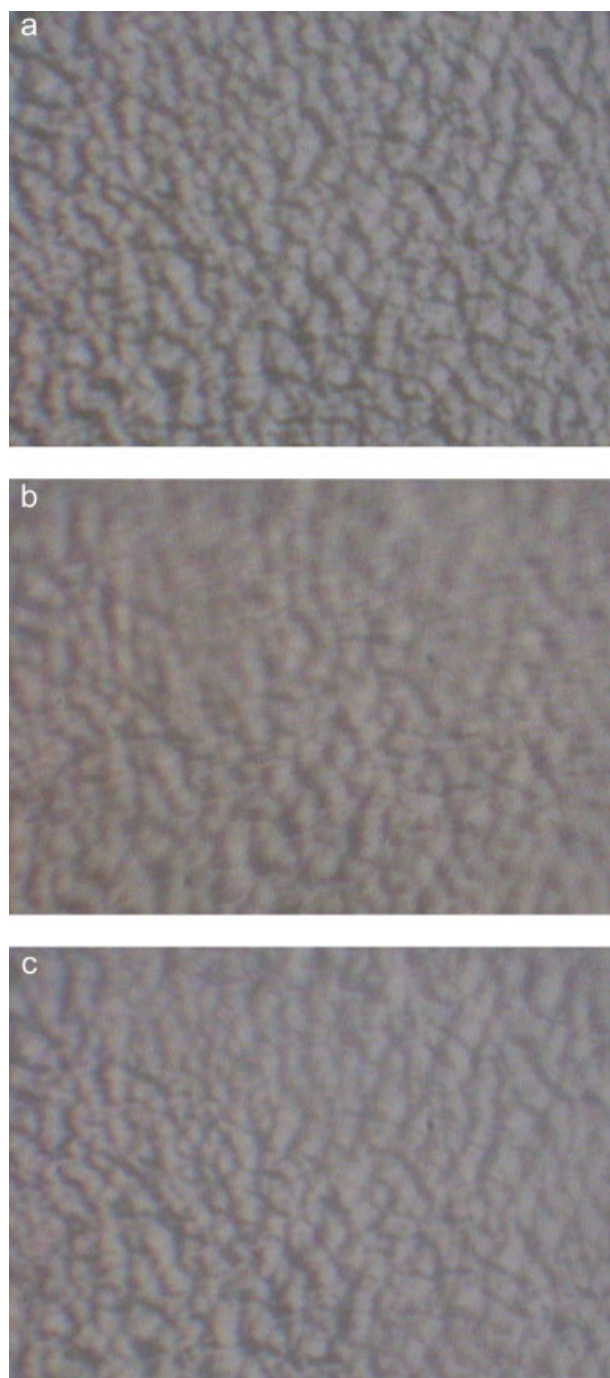


Figure 6 Optical micrographs ($\times 100$) of PMMA/COPE blends (a) HY5, (b) HY10, and (c) HY20. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

COPE. The two phase morphology of PMMA is due to the existence of amorphous and crystalline phase, and the two phase in COPE is due to the presence of hard thermoplastic segment and soft ether-ester phase. Figure 6(a–c) shows the optical micrographs of the HY5, HY10, and HY20 blend, respectively. In Figure 6(a), it is seen that COPE is found to be dispersed as domains in the continuous PMMA matrix.

This is due to the higher melt viscosity and lower COPE content as compared to PMMA in the blend. As the surface of the dispersed phase appears to be smooth which may be due to poor adhesion among the constituents. In Figure 6(b), it is noticed that the surface of the dispersed phase has stretched and become rough owing to increase adhesion between COPE and PMMA. This indicates that at 10 wt % addition of COPE (HY10) in PMMA matrix has very good adhesion among the constituents. This observation supports the increase in impact strength at 10 wt %. In Figure 6(c), it is noticed that both PMMA and COPE phase exists more or less as cocontinuous phase. This may be associated with higher concentrations of COPE and low viscosity of the PMMA phase which may leads to lower the mechanical properties. This observation also supports the decrease in impact strength values for higher COPE content in PMMA matrix.

Effect of chemical ageing

The tensile properties of PMMA and all its blends before and after ageing in different chemical reagents are summarized in Tables II–IV. It is observed that the tensile strength of PMMA after ageing declined about 5–15%, whereas its blends shows increase in tensile strength in the range of 3.6–9.8%. These results indicate that COPE content in the blends enhances the chemical resistance of PMMA for all the compositions. The percentage of tensile elongation of PMMA retains the properties after chemical ageing, but its blends shows reduction in percentage of elongation after chemical ageing. A marginal reduction in tensile modulus after chemical ageing is observed in the case of PMMA, but in the case of PMMA/COPE blends, the tensile modulus has increased marginally or retained after chemical ageing. As the COPE concentration increases, the

TABLE II
Influence of Chemical Ageing on Tensile Strength of PMMA/COPE Blends

Chemical resistance	Tensile strength (MPa) \pm 4%				
	HY0	HY5	HY10	HY15	HY20
Before ageing	60.9	51.9	47.7	44.0	37.8
Ageing in alkali					
5% Na ₂ CO ₃	51.8	53.9	51.4	46.8	40.4
5% NaOH	54.1	54.7	51.6	46.8	40.0
5% NH ₄ OH	53.2	53.8	52.4	47.8	40.2
Ageing in acids					
5% CH ₃ COOH	55.8	55.4	49.9	46.8	40.1
5% HCl	56.2	55.3	51.8	46.9	42.1
5% H ₂ SO ₄	57.8	56.3	52.7	48.2	43.5
Ageing in neutral solvents					
Water	52.3	57.8	52.8	48.9	44.2
<i>n</i> -Hexane	–	53.8	51.9	47.4	38.5

TABLE III
Influence of Chemical Ageing on Tensile Elongation of PMMA/COPE Blends

Chemical resistance	Tensile elongation at break (%) (±4.8 %)				
	HY0	HY5	HY10	HY15	HY20
Before ageing	10.96	37.31	44.22	41.79	35.95
Ageing in alkali					
5% Na ₂ CO ₃	10.40	36.90	37.99	26.71	13.74
5% NaOH	11.70	27.90	34.49	34.07	30.10
5% NH ₄ OH	11.20	28.10	40.36	37.26	26.70
Ageing in acids					
5% CH ₃ COOH	10.80	14.63	14.15	13.70	13.11
5% HCl	12.30	14.27	16.69	29.60	26.60
5% H ₂ SO ₄	12.80	8.39	15.70	15.50	11.90
Ageing in neutral solvents					
Water	12.10	9.66	16.96	14.64	10.20
<i>n</i> -Hexane	12.60	28.78	34.05	31.16	29.00

TABLE IV
Influence of Chemical Ageing on Tensile Modulus of PMMA/COPE Blends

Chemical resistance	Tensile modulus (MPa) ± 4%				
	HY0	HY5	HY10	HY15	HY20
Before ageing	1280	1077	960	895	755
Ageing in alkali					
5% Na ₂ CO ₃	1108	957	943	897	795
5% NaOH	1218	1031	995	925	801
5% NH ₄ OH	1209	1045	1028	948	762
Ageing in acids					
5% CH ₃ COOH	1180	1150	1043	916	902
5% HCl	1208	1093	1033	956	778
5% H ₂ SO ₄	1231	1280	1213	1140	1095
Ageing in neutral solvents					
Water	1201	1395	1040	953	897
<i>n</i> -Hexane	1189	1174	1037	906	857

modulus values increases in comparison with the respective samples before ageing. The tensile behavior results after chemical ageing reveals that the incorporation of COPE into PMMA matrix enhances the chemical resistance of amorphous PMMA material.

The chemical resistant behavior is related with the basic polymer structure, the type of polymer blends, degree of crystallinity, branching, distance between the bonds, energy required to break the bonds, etc., which are to be considered while studying the chemical resistance of plastic materials. The reduction in mechanical properties of PMMA after chemical ageing was expected because of its structural characteristics. But the improvement or retention in tensile strength and tensile modulus of PMMA/COPE blends after chemical ageing may be due to the effect of chemical reagents with the constituents of the blends which helps to improve the interaction among the materials that leads to improve the properties.³⁶ However, the reduction in percentage of elongation after chemical ageing obviously due to the effect of chemical reagents, the materials may undergo physical or chemical changes and hence reduce the elongation.

Effect of heat ageing

The tensile property values of virgin PMMA and all its blends before and after heat ageing at 80°C for

168 h is given in Table V. Tensile properties of PMMA reduce after heat ageing as expected. But in the case of all its blends after heat ageing, tensile strength and tensile modulus has increased marginally and tensile elongation decreased as expected. These results indicates that the presence of COPE resist heat ageing and stabilizes PMMA. Plastics being a heat sensitive material, when exposed to heat may be subjected to many types of physical and chemical changes. The effect of heat and its duration determines the extent and type of changes. The more time exposure at elevated temperatures may generally cause thermal degradation which may leads to progressive changes in physical properties. Heat ageing generally relieve molding stress and becomes more compact and increase crystallinity or even brittleness which may lead to higher stiffness and mechanical properties. However, for a long period of heat ageing under elevated temperatures, all plastics get degradation.

CONCLUSIONS

Blends of PMMA with thermoplastic COPE under different compositions (5–20 wt % COPE) were prepared using twin screw extruder. The physicochemical, thermal properties, surface morphology,

TABLE V
Influence of Heat Ageing on Tensile Properties of PMMA/COPE Blends

Blend designation	Tensile strength (MPa) ± 4%		Tensile elongation at break (%) ± 4.7%		Tensile modulus (MPa) ± 4.4%	
	Before ageing	After ageing	Before ageing	After ageing	Before ageing	After ageing
HY0	60.9	54.8	10.96	8.20	1280	1216
HY5	51.9	52.2	37.31	6.85	1077	2529
HY10	47.7	58.1	44.22	8.18	960	2297
HY15	44.0	50.7	41.79	14.38	895	2342
HY20	37.8	44.6	35.95	8.50	755	1967

and chemical/heat ageing resistance were investigated and compared with virgin PMMA. The mechanical properties of the blends are found to be significantly influenced by the blend compositions. A significant improvement in impact strength and percentage elongation at break was observed for all the PMMA/COPE blends as compared to virgin PMMA. The impact strength of PMMA is increased from 19.1 to 42.89 J/m by the addition of 10 wt % of COPE. However, tensile strength and tensile modulus has reduced marginally in all the blends. Optical micrograph studies reveals that two phase morphology for PMMA/COPE blends, in which COPE is dispersed uniformly through out the PMMA matrix. Various composite models have been used to fit the experimental mechanical data. The tensile strength predicted by the series model and tensile modulus predicted from Kerner's model were found to be very close to the experimental results. Chemical and heat ageing resistance of PMMA has been significantly improved by the incorporation of COPE which indicates that COPE stabilizes PMMA from the deterioration by the chemical reagents and heat ageing.

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