Studying of The Impact Strength of Rubber- Toughened Thermosets

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Abstract

In this investigation, the development of composite materials was achieved in two steps. The first, by the hybridization of the resin matrix with different elastomers to prepare binary polymer blends, these blends have been synthesized by mixing either of (Nitrile Rubber(NBR), Styrene Butadiene Rubber(SBR) and Butadiene Rubber(BR)) with the Epoxy resin(EP) and Unsaturated polyester resin(UP). The second step is reinforcement of the polymer blends with 30% of two types of fibers. The first, is by using E-glass fibers alone while the second by utilizing a hybrid of E-glass fibers with Kevlar fibers (type-49). Different weight percentages (5, 10, 15,…., 50)% of those elastomers were mixed with each of EP and UP. The optimum mixing ratios (OMR) of those blends have been selected depending on achieving acceptable macro miscibility, best adhesion between two phases and highest impact strength (I.S) of the resulting blends. The values of (I.S) increased gradually with increasing of rubber content until its maximum value (OMR), and then it goes down as the percentage of the elastomer increases. Five blends with (OMR) were prepared : EP/NBR(80/20)% , EP/BR(70/30)% , UP/NBR (90/10)%, UP/SBR (90/10)% , UP/BR (90/10)%, while a phase separation of SBR rubber from EP resin has taken place at all ratios; then, these blends were reinforced with single and hybrid fibers. Impact test was carried out at different environmental conditions to have full description about the mechanical behavior of those blends and their composites under effect of the impact stress.

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دراسة مقاومة الصدمة لراتنجات متصلدة حرارياً مقواة بالمطاط

الخلاصة

في هذا البحث ، تم تطوير المواد المتراكبة على مرحلتين ، الاولى بتهجين الاساس الراتنجي بمواد مرنة مطاطية مختلفة لتحضير خلائط بوليمرية ثنائية والتي حُضرت بواسطة خلط كل من انواع المطاط الثلاثة (BR , SBR , NBR) مع راتنجي الايبوكسي والبولي استر غير المشبع . اما المرحلة الثانية فتضمنت تسليح تلك الخلائــــط البوليمرية بنوعيـــــن من الالياف وبكســـــر حجمــــــي قـــــدره (30%) . أُستخدمت طريقتين للتسليح الاولى باسخدام الياف الزجاج بمفردها والثانية بواسطة استعمال هجين من الياف الزجاج والكفلر نوع (49) . **وقد أُستخدمت نسب وزنية مختلفة لكل نوع من المطاط شملت** %(**5,10,15,.....,50**). أذ **تم** خلطها مع راتنج الايبوكسي (EP) مرة وراتنج البولي استر غير المشبع (UP) مرة اخرى ، فاختيرت نسب الخلط المثلى (OMR) اعتماداً على استحصال امتزاجية مناسبة ، افضل حالة التصاق بين الطورين ومقاومة الصدمة (I.S) الاعلى
</div>

للخلائط الناتجة وبعد البحث والدراسة وجد بان قيم (I.S) تزداد تدرجياً مع زيادة المحتوى
المطاطي لحين الوصول الى قيمة (OMR) ومن ثم تبدأ بالتناقص عند زيادة نسبة المطاط
المحتواة في الخليط. وقد تم التوصل الى خمسة خلائط وبنسب خلط مثلى (OMR) هي :
%(80/20) لخليط EP/NBR ، %(70/30) لخليط EP/BR و %(90/10) للخلائط
UP/NBR ، UP/SBR ، UP/BR في حين حصل انفصال طوري واضح لمطاط SBR
من راتنج الايبوكسي (EP) عند جميع نسب الخلط ، ومن ثم سُلحت تلك الخلائط المثلى
بالياف منفردة وهجينة . وقد أجري اختبارالصدمة عند ظروف بيئية مختلفة للتوصل الى
وصف متكامل عن السلوك الميكانيكي لتلك الخلائط ومتراكباتها تحت تأثير الاجهاد الصدمي .

Introduction

The use of polymer blends and alloys has grown so fast compared with other polymeric materials system mainly because of their low cost and their acceptable performance [1].

Polymer materials are made in different varieties; the polymer blends are classified as one of those varieties. Polymer blends are made by physical mixing of two (or more) different polymers, to produce a mixture that has desirable mechanical properties. This type of polymers is used widely to produce industrial fibers, in some cases, textile fibers are fabricated from mixing of different polymers [1].

There are many reasons why polymer blending is one of the most important areas in polymer research and development. Among these reasons, the most important is, perhaps, that polymer blends offer a fast and cheap way to obtain new polymeric materials. These materials generally exhibit a range of properties which varies between the properties of their components. Moreover, their properties may be complementary and difficult to be found together in the case of a single component [2,3].

There has been a great deal of interest in the studies of the structure and properties of polymer blends. The practical motivation for this interest is the achievement of desirable mechanical, rheological or other properties. Because of the generally immiscible nature of polymers, polymer blends usually exhibit microphase separations with two-phase structure [4].

Bing et.al.[5] investigated the microstructure, mechanical properties and fracture behavior of liquid rubber toughened thermosets. Phenolic epoxy resin was toughened by carboxyl-randomized butadiene acrylonitrile copolymer (CRBN) for use as composite matrix. By adding different parts of butadiene acrylonitrile copolymer (BN-26, without carboxyl contained) to CRBN, different sizes of rubber domains and different numbers of chemical bondings between the resin matrix and the rubber phase are obtained. They found that small rubber particles are cavitated during the crack development. They concluded that the phase separation of (CRBN) from phenolic epoxy resin can be promoted by adding a small amount of BN-26.

A group of authors[6] carried out the dielectric and morphological investigations into phase separation and cure in rubber-modified epoxy resins. The rubber modifier used was a carboxyle-terminated butadiene acrylonitrile (CTBN). The mixture was initially homogeneous, but after a short period of time, it underwent phase separation and this process was marked by the appearance of a dielectric peak associated with ion-charge migration

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within the occluded rubbery phase. Under Scanning Electron Microscope (SEM), a phase separated morphology was observed consisting of spherical rubber particles dispersed in an epoxy matrix.

Blends consisting of ductile polycarbonate (PC) and brittle poly methylmethacrylate (PMMA) polymers have been prepared by Tjong and Xu [7]. The mechanical properties of the blends were studied by tensile and impact testing. The (Tg) of the (PC) and (PMMA) phases tend to shift towards each other for the (PC) rich blends, indicating that (PC) and (PMMA) are partly miscible in these blends.

Ebling et.al. [8] studied the effect of acrylonitrile (AN) content on the adhesion of poly(styrene-co-acrylonitrile,SAN) to polycarbonate (PC) by measuring the delamination toughness of (PC/SAN) microlayers in the T-peel test. It was concluded that if the craze initiation condition is lower than the interfacial toughness; formation of a craze zone increases the delamination toughness dramatically.

The effect of chain microstructure on adhesion of ethylene copolymers to polypropylene (PP) was studied by Poon et.al.[9] through using coextruded microlayers. The adhesion was measured by delamination toughness (G) using the T- peel test , and interfacial morphology was viewed by atomic force microscopy. Good adhesion to PP was achieved with homogeneous metallocene catalized copolymers (mPE) with density 0.90 g. cm⁻

3 or less. Good adhesion was attributed to entanglement bridges.

Meanwhile, Tayyab , Ibnelwaleed [10] investigated the influences of short chain branching (SCB) on the melt miscibility of low-density polyethylene (LDPE) with linear high-density polyethylene (HDPE) through dynamic and steady shear measurements carrying out by rheological methods. The level of (SCB) in LDPE was found to have a strong influence on its miscibility with linear HDPE.

Geum [11] investigated the influence of liquefied wood (LW), melt index (MI) and polymer on the mechanical properties and creep behavior of liquefied wood-polymer composites(LWPC) Polymer and LW showed significant effects on the flexural strength properties. MI showed a significant effect on the creep deflection. The overall creep deflection was not affected by the addition of 10% LW.

Soderholm [12] reports that many of the polymers used in composites,
including EP and UP take up several percents of water upon immersing them in it, which causes those materials to swell and plasticize.

The aim of this research is to modify the mechanical and physical properties of thermoset resins to reduce their brittleness by mixing them with elastomers. Epoxy (EP) and Unsaturated polyester (UP) resins have been selected as a brittle materials, while NBR, SBR and BR were chosen as elastomers.

Experimental part
The Materials Used:
(1) Resins
Two different types of the resins were used in the current study as follow:
(a) Epoxy Resin (EP)
Epoxy resin (type Conbextra EP10) was used in this research; it is a liquid with moderate viscosity and capable to be converted to solid state by adding the solution (Metaphenylene Diamine,

MPDA Jordon Co.) as hardener. This hardener is
a light liquid with yellowish color, the ratio of this hardener to the epoxy is about (1:3).

(b) Unsaturated polyester resin (UP)
It is a liquid with moderate viscosity which can be cured to the solid state by adding (Methyle Ethyle Ketone Peroxide,MEKP," Jordon Co.") as a hardener, while cobalt octoate acts as a

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catalyst to accelerate the solidification process. The percentage of the hardener to the resin is (2%) while it is (0.5%) for accelerator.

(2) Elastomers
Three different types of rubber were used in this research (SBR, NBR and BR "Jordon Co."). Normally these types of rubbers are handled as large solid pieces,

(3) Fibers
The following two types of fibers have been used in this investigation:

a- E-glass fibers:- It is well known, that E-glass has several shapes, the used shape was chopped strand mat (CSM). This mat was cut to form the dimension that would fit the area of the mold.

Preparing of Polymer Blends and Composites:

In this work, mechanical mixing was used to prepare binary polymer blends from (EP or UP) resins with different percentages and types of rubbers (NBR, SBR, BR).
EP resin was mixed with (5, 10, 15,…, 50) % either of the above mentioned rubbers. The optimum mixing ratios (OMR) were selected based on acceptable macromiscibility, best adhesion between the two phases was noticed by the optical microscope and highest impact strength for each blend during the examination. The same procedure for (UP) resin was applied.
To develop the properties of blends that have the (OMR), they were reinforced by E-glass fibers alone firstly, then in combination with Kevlar fibers (hybrid). All the samples of composites were prepared with the same volume fraction
composites were prepared according to the following procedure:
1- Clean galvanized metal mould with dimensions (250*250*30) mm^3 was used for casting the sheets of the blends. Polymer sheet was fixed on the inner mould faces before casting to facilitate the releasing of casting blends and having smooth faces.

so they have to be cut into smaller ones. Toluene is required to dissolve each of these rubbers in small glass sealed containers. The weight of the used solvent is equal to twice of that of rubber (2 solvent / 1 rubber). The properties of the elastomers used in this study are listed in Table (1).

b-Kevlar fibers:- This fibers are produced by Du Pont Co. with different trade marks (29, 49 and 129). The one that was used in this work was type (49). Table (2) shows some of the properties of the two above-mentioned types.

ratio of the fibers ((Φ =30%)) for both cases which was calculated by applying the relationship:

$$\Phi = \frac{1}{1+ ((1-\psi)/\psi)*(\rho_f / \rho_m)} \quad …..(1)$$

where Φ, ψ are the volume and weight fractions of the fibers respectively.
ρ_f , ρ_m are the density of fibers and matrix respectively.
The density of the prepared blends was determined from the equation:
$\rho_m = x_1\rho_1+x_2\rho_2$………….(rule of mixtures),
where ρ_m : the density of the matrix (polymer blend).
ρ_1, ρ_2 : the density of the first polymer and the second respectively.
x_1, x_2 : the percentages of the first polymer and the second respectively.

The above mentioned blends and
2- Cover plate, with identical dimension of the mould face, was used to apply appropriate load on the casting sheet for releasing bubbles, to have a specified thickness and smooth face.
3- Casting sheet was left inside the mould at room temperature about (24h) for (EP) blends and about (3hour) for (UP) blends.

4- After solidification, the casting sheets were released from the mould and placed in an oven with (50°C setting temperature) for (3h) to post cure the considered sheets.

5- The testing samples were obtained by cutting the casting sheets according to (ISO-179)

Identical procedure was used for preparing composite samples (hand lay-up technique) adding to that, E-glass and kevlar fibers were cut and aligned in the mould, before pouring the blend. Depending on the required composite thickness, certain numbers

Impact test instrument

Charpy impact test consists of standard test piece that would be broken with one flow of a swinging hammer. The test piece is supported at both its ends in a way that the hammer strikes it at the middle [17].

Impact strength (I.S) is calculated by applying the relationship:

I.S = Uc/A ....(2)

where **Uc**: is the fracture energy (Joule) which is determined from charpy impact test instrument.

of layers were stacked until the desired thickness was obtained.

The single laminated composite contains only E-glass fibers as reinforcing material, while the hybrid laminated composite contains Kevlar fibers in addition to E-glass fibers as reinforcing material). Fig.(1) shows that the outer skins of the hybrid composite includes Kevlar fibers only (Φ=15%) while the core contains layers of E- glass fibers (Φ=15%).

A: is the cross-sectional area of the specimen.

Where the impact test was carried out, the samples were subjected to optical microscope examination to notice the fracture morphology of these samples.

This test was conducted in different conditions (air and water) at room temperature; the immersion process in water was carried out by soaking all the samples in distilled water into glass containers for three months.

Experimental results and discussion

The conventional charpy impact test is used to evaluate the impact strength of the blends that have (5, 10, 15, 20, 25, 30, 35, 40, 45, 50)% of NBR, SBR and BR in either of EP and UP. Fig.(2) and Fig.(3), which show the effect of rubber content on the impact strength values of the prepared blends and composites. The presence of the second phase (rubber) increases the energy required to maintain a given crack- growth rate of the sample and therefore the impact strength increases of these (resin-rubber) blends.

Fig.(2) and Fig.(3) clearly demonstrate the effect of blending (NBR, SBR and BR) with EP and UP respectively on the impact strength of the resulting blends. In general, it is well known that the cured rubber-modified epoxy exhibits a two – phase microstructure which consisting of relatively small rubbery particles [18]. These particles are dispersed into the matrix of epoxy and the resulting phase separation nature will be related to the percentages of the rubber and the methodology of the preparation. Any increase in the rubber amount in the matrix

would lead to an increase in the average size of the dispersed phase particles and eventually failing to produce a compatible and applicable blend [19].

The above-mentioned phenomenon is clearly noted in the present work, it can be seen that when the percentages of (NBR) rubber in the (EP/NBR) blend increases to more than 20%, (Fig.2), the rubber phase has notably separated from the epoxy leading to decrease in the impact strength of the blend. The same phenomenon has happened to (BR) rubber in (EP/BR) blend but in a different percentage (30%). When EP is mixed with any percent of SBR, it forms heterogeneous system due to coalescence of SBR particles and a discrete

The (UP/rubber) blends has exhibited compatibility but at lower ratios and demonstrated phase separation behavior similarly to that of (EP/rubber) blends at ratios above 30% of rubber. Although this behavior is well proved experimentally, From Fig.(2), the Impact strength of (EP) can be maximized to almost (32, 10.4) KJ/m^2 on addition of (20, 30) wt% of NBR and BR rubbers respectively. From Fig.(3), the impact strength of (UP) could be maximized to almost (15.5, 10, 31.4) KJ/m^2 on addition of about (10%) of NBR, SBR and BR rubbers. If the percentage of elastomers in the brittle matrix increased to more than the above mentioned percentages, the impact strength would decrease to lower values. This indicates that the impact strength of binary blend is not simply an additive, and its dependence on blend composition reveals the influence of blend morphology, state of dispersion

From Fig.s(2) and (3), the optimum mixing ratios (OMR) of NBR and BR in EP resin are 20 and 30 wt% respectively. while the (OMR) of NBR, SBR and BR in UP resin is 10 wt%. Those blends with (OMR) are presented in Table(3) and they are going to be considered as a basis for further investigation.

phase embedded in EP phase. This may be attributed to the weakness of the interfacial forces between the EP and SBR. It is known that EP is recognized by its very high polarity [20] , while SBR has no polarity and it is recognized by the presence of aromatic rings [14] . A mixing of such two polymers would exhibit a steric hindrance, which prevents the formation of any physical bonding between them. Fig.(4) shows the optical microscopy image of EP/SBR mixture, which explains the morphology of such system. From this image, it is quite clear that SBR is almost completely discrete from the mother matrix, which means the impossibility of forming a compatible and applicable blend from these two components. but there is no decisive explanation for it, there are only some ideas or expectations which relate this behavior to many factors such as composition, degree of homogeneity, crystallinity and interfacial adhesion[3,21,22]

and any other structural parameter on impact toughening of this blend. Occurrence of a maximum in the impact strength at a particular blend composition may be attributed to the critical size and geometry of dispersed phase domains. Chiang and Song [22] have suggested that the rubber particles are often spherical with spherical inclusions of the brittle phase. At concentrations larger than the related maximum value of the rubbery phase, the dispersed particles tend to agglomerate or to form elongated rather than spherical particles which reduce the impact strength[21].

All the blends with (OMR) were reinforced in two ways, the first by using 30% of E-glass fibers alone, while the second by using a hybrid of E-glass and Kevlar fibers at the same volume fraction. Table (4) shows an increasing of I.S due to the presence of those reinforcing materials. The reason behind this increase, is that the

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fibers act to distribute the stresses on larger volume of the material instead of localizing those stresses [16,23]. The increase of I.S of hybrid/composites is more than that of E-glass/composites because Kevlar fibers act as body armor with higher toughness. It is essential to mention that the I.S of E-glass fibers and Kevlar fibers are 160 KJ/m^2 and 316 KJ/m^2 respectively [24], which imply why the composites with Kevlar fibers have greater I.S.

Figures (5) and (6) are photographic images showing the fracture zone for the tested composites, which reflect the brittle fracture mode with complete division for E-glass composite, and the ductile plastic deformation mode with partial division for hybrid composite respectively [16].

It is accepted that during application, the surrounding environments normally affect most blends and composites, particularly humidity. The reason behind that is the possibility of permeating water molecules into those materials, which may lead to a reduction in the mechanical properties by weakening the bonds between the two phases of the blends or between the fiber/matrix interface [25].

The blends and composites under test were immersed in water for three months at room temperature. The impact strengths of those samples were measured every 15 days and the results are displayed in Figures (7) to (10).

The general external appearance of the immersed materials reveals that, the soaked specimens do not undergo any swelling, but they are plasticized. The same above-mentioned Figures show that absorbed water has limited effect on the impact strength of materials. This indicates that those materials are sufficient to be used in humid conditions without significant deterioration in their I.S.

Conclusions:
Based on experimental results, which are presented in this work, the following conclusions can be drawn:-

1- NBR, SBR and BR rubbers are an excellent impact modifier to (UP) when they are added in specified percents (10% for all of them) as they act to reduce the brittleness of the resin.

2- SBR can not be used as an impact modifier for (EP) but NBR and BR do,when they are added in limited ratios (20% for NBR and 30% for BR),because SBR is almost completely dispersed in the epoxy resin, whichmeans the impossibility of forming a compatible and applicable blend from these two components.

3- When the rubber content of the blend increases, the impact strength increases too and then gradually decreases where phase separation between the two phases begins to take place.

4- The morphological and impact strength of the elastomeric - modified resin depend strongly on the volume fraction (percentage) of the second phase.

5- The laminated composites that are composed of hybrid of fibers (E-glass and Kevlar) are recognized by their light weight and their higher impact strength when they are

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compared with the composites of E- glass fibers alone.

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Table (1): Some properties of the used elastomers[13,14,15]

Rubber	Density (gm/cm³)	Tg (ᵒC)	Tensile Strength (MPa)	Elongation (%)
NBR	1	-22	20.68	650
SBR	0.94	-60	20.68	450
BR	0.91	-85	24.13	550

Table (2): Properties of E – glass and Kevlar (49) fibers [16].

Fiber	Density (gm/cm³)	Diameter (µm)	Tensile Strength (GPa)	Young's Modulus (E,GPa)	Fracture Strength (σ,GPa)
E-glass	2.6	11	3.33	76	2.0
Kevlar49	1.45	12	3.62	130	3.0

Table (3): The optimum mixing ratio (OMR) of NBR, SBR and BR in
EP and UP.

blend	Optimum Mixing Ratio (OMR)
EP/NBR	(80/20)%
EP/SBR	Failed at all percentages
EP/BR	(70/30)%
UP/NBR	(90/10)%
UP/SBR	(90/10)%
UP/BR	(90/10)%

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Table (4): Impact strength of the materials before and after the
reinforcement

Specimen	Impact Strength (I.S), KJ/m^2		
	blend	blend +E-glass fibers (G.F)	blend + hybrid fibers (H.F)
EP/NBR(80/20)%	31.91	170.76	179.11
EP/BR (70/30)%	10.41	153.57	185.35
UP/NBR(90/10)%	15.52	113.88	172.24
UP/SBR (90/10)%	10.01	92.55	185.13
UP/BR (90/10)%	31.36	99.33	194.97

(b)
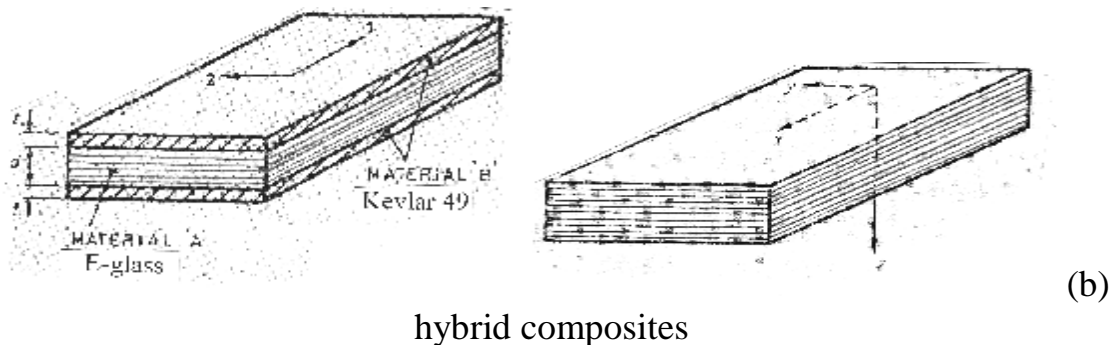
hybrid composites
Fig.(1): Schematic drawing of the layers in the composites. (a) single,
.
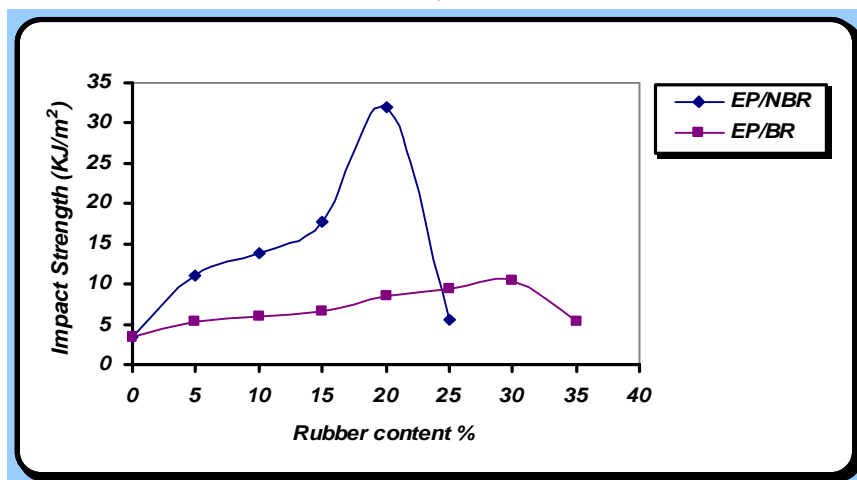
Fig.(2): Charpy impact strength variation with NBR and
BR content in EP resin.
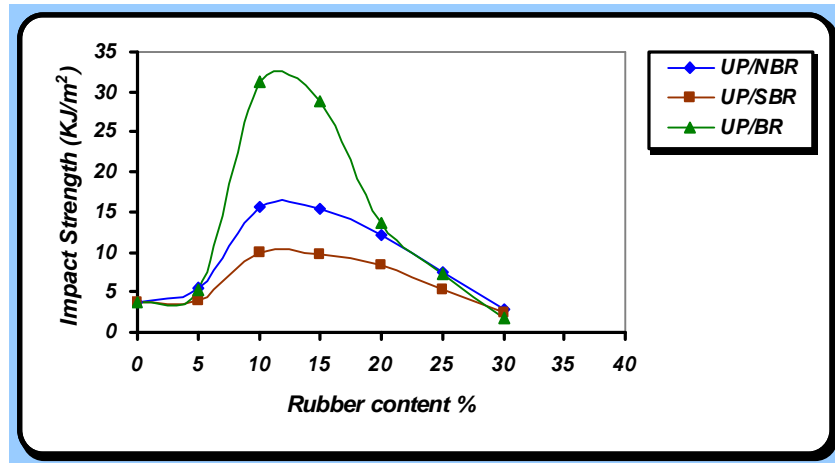
Fig.(3): Charpy impact strength variation with NBR,
SBR and BR content in UP resin
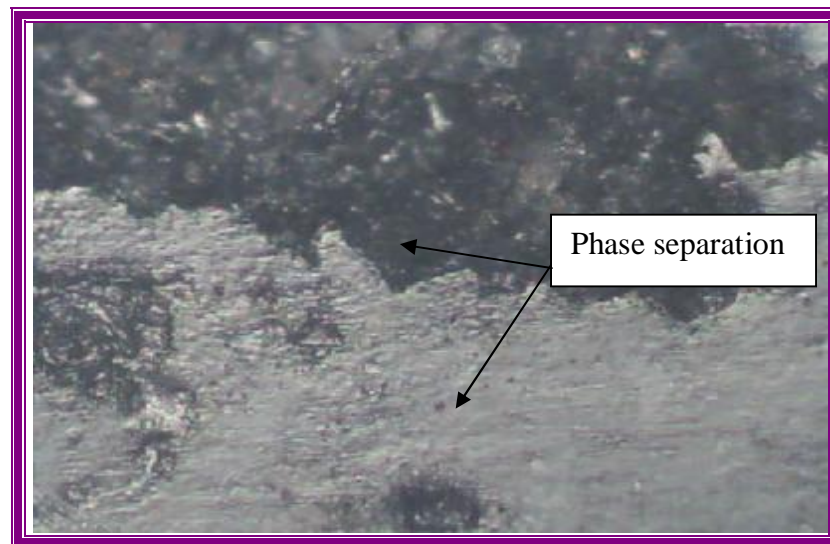
Fig. (4): Optical micrograph shows phase separation of (EP/SBR)
blend; the dark region is EP while white region is SBR

Fig.(5): Impact fracture zone of E-glass composite.
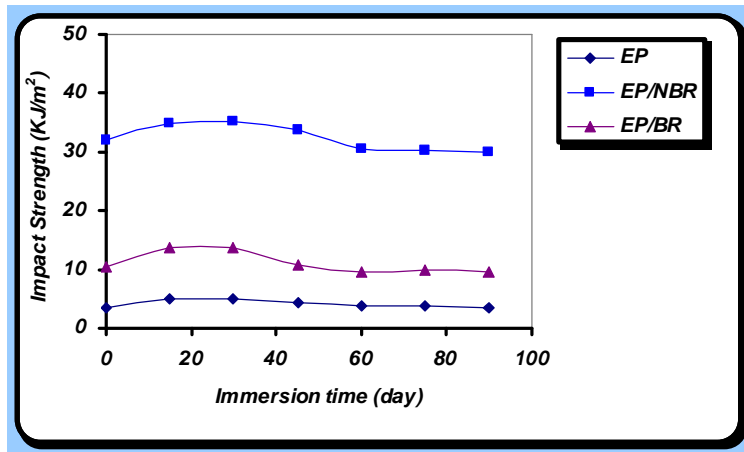
Fig.(6): Impact fracture zone of hybrid composite

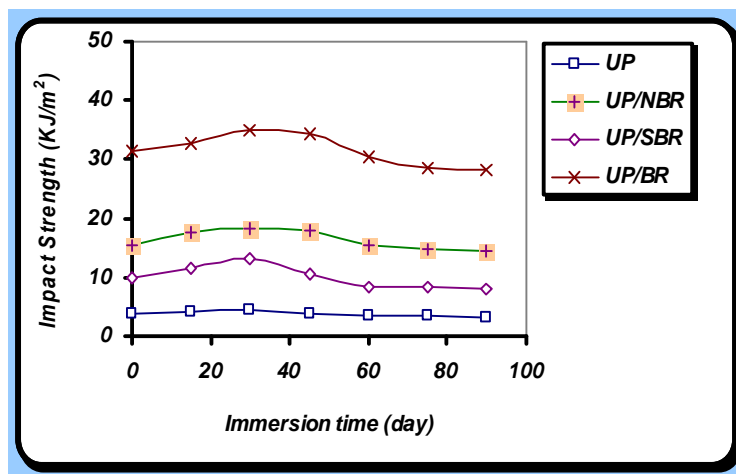Fig.(7): Water effect on values of the
(I.S) of EP and their blends.

Fig.(8): Water effect on values of the (I.S)
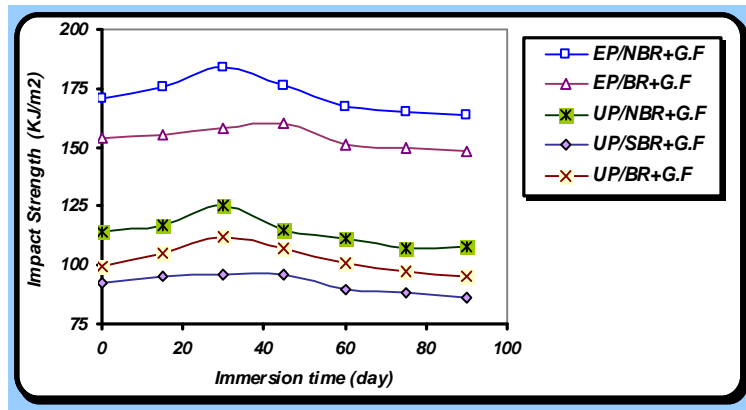of UP and their blends.

Fig.(9): Water effect on values of the (I.S) of
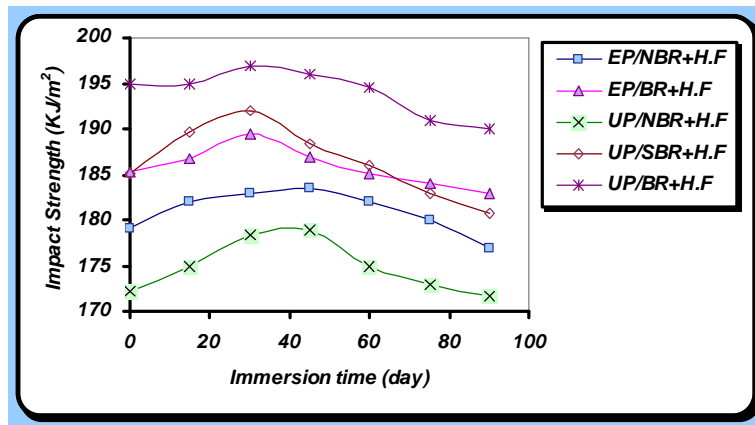E- glass composites(G.F).

Fig.(10): Water effect on values of the (I.S) of
hybrid composites (H.F).