

STUDYING THE EFFECT OF NANO CARBON BLACK ON MECHANICAL PROPERTIES OF UNSATURATED POLYESTER RESIN

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ABSTRACT

The recent emergence of nano science and nano technology has added another dimension to the staple of the modern composite technology. Instead of micron diameter fibers and particles, nanoparticles are being incorporated into polymer matrices to form a composite known as polymer nano composites (PNCs). In this study, the composites were prepared from unsaturated polyester and different weight percentages of carbon black (0, 3, 6, 9 and 12 %) nanoparticles using hand lay out. The mechanical properties – such as tensile strength, modulus of elasticity, elongation at break, hardness, bending and impact properties were studied. The results show that the tensile strength, modulus of elasticity, impact strength, fracture toughness, flexural strength and maximum shear stress was achieved at (3wt. %) of carbon black, while elongation at break decreases with an increasing of carbon black weight percentage. The hardness of composite increased with the increasing of carbon black weight percentage.

KEYWORDS: Unsaturated polyester, Carbon black, Mechanical Properties, Impact, Bending, Flexural, Shear stress.

دراسة تاثير اسود الكربون النانوي على الخواص الميكانيكية لراتنج البولي استر الغير مشبع

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الخلاصة

ان الظهور الحديث لعلوم وتكنولوجيا النانو قد اضاف بعدا آخر في تكنولوجيا المواد المتراكبة الحديثة. حيث يجري إدماج دقائق ذات حبيبات متناهية في الصغر (نانوية) في المادة الاساس البوليمرية بدلا من ألياف ودقائق قطرها ميكرون ، لتكوين مادة متراكبة تعرف باسم مواد متراكبة نانوية (PNCs). في هذا البحث، تم تحضير مادة متراكبة من البولي استر الغير المشبع وتم اضافة اسود الكربون ذي الحجم النانوي بكسور وزنية مختلفة (0 ، 3 ، 6 ، 9 ، 12 %) باستخدام طريقة القولبة اليدوية. حيث تم دراسة الخواص الميكانيكية مثل اجهاد الشد ، معامل المرونة، الاستطالة عند الكسر، الصلادة، خصائص الانحناء والصدمة. حيث أظهرت النتائج بأن أعلى قيمة لاجهاد الشد، معامل المرونة ، مقاومة الصدمة، متانة الكسر، مقاومة الانحناء والحد الأقصى لاجهاد القص تحدث عند نسبة (3%) من اسود الكربون، في حين ان الاستطالة عند الكسر انخفضت مع زيادة الكسر الوزني لاسود الكربون. ولوحظ كذلك ان صلادة البولي استر الغير مشبع تزداد مع زيادة الكسر الوزني لاسود الكربون.

INTRODUCTION

Today, composite materials are quite common and are used in nearly every segment of civilian, military industries, automotive, construction and packaging application. The major advantages of composite materials are low density, high specific strength and stiffness, good corrosion resistance and improved fatigue properties. Because of these properties, they have successfully replaced many conventional metals [Wafaa 2011 and H.P.S. Abdul Khalil 2010].

For ages, polymers were reinforced with a variety of micron scale particles to form polymer composites. These particles which include silicates (mica, talc, silica and fibre glass), metal oxides (titania, alumina), calcium carbonate and carbon black (CB), were successfully used as additives or reinforcements to improve various properties of polymers [Tony Nguyen 2003 and C.S. Obayi 2008].

Polymer nanocomposites consist of a polymer matrix and filler, having at least one of its dimensions below 100 nm. The first reports were published in the eighties and early nineties of last century. Ever since, various aspects of polymer nanocomposites have been studied. The value of nanocomposites lies in the fact that the transition from macro/micro size particles to nanoparticles results in dramatic changes in the physical properties of the same element. Since many physical and chemical properties of materials and their interactions with other materials are guided by their surface properties, and as nanomaterials have a huge surface area per unit volume, the ability to tinker and control nanoparticles within polymer matrices taken on high importance [Siva Subramanyam Movva 2010 and Steven A. Edwards 2006].

Polymer nanocomposites comprise a new class of materials in which nanoscale particulates (e.g., clay or other inorganic minerals) are finely dispersed within the matrices and intensive research efforts have been devoted to the development of nanocomposites. It is known that the mechanical properties of the composites are, in general, strongly related to the aspect ratio of the filler particles. The mechanical properties of the composites filled with micron sized filler particles are inferior to those filled with nanoparticles of the same filler. In addition, the physical properties, such as surface smoothness and barrier properties cannot be achieved by using conventional micron sized particles [R. Baskaran 2011, Yizhong Wang 2000 and Kemal 2009].

Unsaturated polyester resins (UPRs) are one of the most widely used thermosetting materials because they are relatively inexpensive and offer advantages such as being light in weight and possessing reasonably good mechanical properties, having tremendous versatility and low cost. The use of UPRs in bulk and sheet molding compounds results in composite materials that have high strength, dimension stability, and very good surface qualities. They have many applications in automotive, aircraft, electrical, and appliance components as substitutes for traditional materials. There are various types of nanoparticles and they come in various sizes and shapes. Common particle geometries are shown in **Fig. 1** broadly they are classified into particulate or spherical particles, fibrous, and layered particles. Carbon black, silica nanoparticle, and other metallic nanoparticles fall under the particulate materials category [Siva Subramanyam Movva 2010, R. Baskaran 2011 and Yano. K 1993].

Carbon black is the general term used to describe a powdery commercial form of carbon; a lot like graphite, Carbon black is virtually pure elemental carbon in the form of colloidal particles that are produced by incomplete combustion or thermal decomposition of gaseous or liquid hydrocarbons under controlled conditions [Barry park 2007 and International Carbon Black Association 2004]. Carbon black used principally for the reinforcement of polymer, as a black pigment, and for its electrically conductive properties. It is a fluffy powder of extreme fineness and high surface area, composed essentially of elemental carbon. Carbon black is one of the most stable chemical products. In a general sense, it is the most widely used nano material, with its aggregate dimension ranging from tens of nanometers to a few hundred nanometers, and imparts special properties to composites of which it is a part [M. J. Wang

2002]. Carbon blacks are categorized, on the basis of different production processes by which they are made, as acetylene black, channel black, and furnace black, lampblack or thermal black over 95 % of all carbon black produced today is furnace black. ASTM designations, the older nomenclature, particle size, surface area, and structure of some blacks are given in **Table 1** [Y. k. peng 2007, V. O. Jobando 2006 and C. A. Harper 2006]. Many investigations about the interest of nanoparticles such as alumina, silica [R. Baskaran and Petrovicova R 2000], zinc oxide [Bermudez M-D 2010], calcium carbonate [Chi-Ming Chan 2002], Nano clay [N.Rajini 2012]. have been used to prepare nanocomposites. but only few works concern use of unsaturated polyesters filled with carbon black nanoparticles.

The aim of this work is to prepare a nano composite for unsaturated polyester reinforced with carbon black and studying its mechanical properties.

EXPERIMENTAL WORK

Materials Used

- ❖ **Unsaturated polyester resin (UP)** supplied by (SIR) company, Saudi Arabia were used in this work. 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 catalyst to accelerate the solidification process. The percentage of the hardener to the resin is (2%) while it is (0.5%) for accelerator. Which has properties as shown in **Table 2**.
- ❖ **Carbon black (N220) with particle size 23 nm** used in this research come from Al Dewania factory for tire manufacture, Iraq. Carbon black (N220) used in this work Which has properties as shown in **Table 3** [www.konimpex.pl 2005]:

Composite Fabrication

- The carbon black was dried in an oven at a temperature of 200°C for 12 hours.
- The masses of the reinforcement (Carbon black) and Matrix (unsaturated polyester) materials were calculated according to the role of mixture.
- Prior to mixing, cobalt accelerator was added to the unsaturated polyester resin and the moulds were uniformly rubbed with mould release agent and dried.
- The composites were prepared with (0, 3, 6, 9, and 12%) weight percentage of carbon black. Carbon black was added carefully and gradually to the solution of polyester resin to avoid the loss of carbon black and to avoid bubbling during mixing. The composite mixture was stirred and observed to ensure that the carbon black mixed well with the matrix or until a homogeneous mixture was obtained.
- The mixture was poured from one corner into the mould (to avoid bubble formation which causes cast damage) and the uniform pouring is continued until the mould is filled to the required level.
- The mould was placed on an electrical vibrator to remove any residual bubbles (i.e. captured gas that evaporates during mixing and temperature rise) and to guarantee the distribution of the cast inside the mould.
- The mixture was left in the mould for (24) hrs at room temperature to solidify. Then the cast was aged for one week to dry off. This step was important to reveal complete polymerization, best coherency, and to relieve residual stresses.
- The specimens were cut according to the standard dimensions for each test, using different cutting tools.

CHARACTERIZATION

Tensile Tests

The tensile properties (modulus of elasticity, tensile strength and elongation at break) of the composite were tested according to ASTM D638 for different samples at 20°C, with the use of dumbbell shaped samples as shown in figure (2). The applied force was (1KN) with a pulling device velocity (5 mm/ min). The tests being carried out using the microcomputer controlled electronic universal testing machine Model (WDW-50E).

Hardness Tests

In this work the hardness is done by using shore D according to ASTM D 2240. Five measurements of hardness were made at different positions on the specimens. The specimen should be at least 6 mm in thickness, the surface on which the measurement was made had to be flat and the lateral dimension of the specimen had to be sufficient to permit measurements at least 12 mm from the edges as shown in **Fig. 3** [Standard Test Method for Polymer Properties 1988].

Bending Test.

The bending specimens was prepared according to (ASTM D- 790) with the dimension of (4.8*13*191) mm and (span to depth) ratio of 1:32. flexural strength and maximum shear stress (τ_{\max}) were obtained mathematically from equations (1 and 2) respectively [Annual book of ASTM 1989].

$$F.S = \frac{3P\ell}{2bd^2} \quad (1)$$

$$\tau_{\max} = \frac{3P}{4bd} \quad (2)$$

Where:

p: load at fracture (N).

d: thickness of specimen (m)

b: width of specimen (m)

Impact and fracture toughness test

This test is performed according to (ISO-197) at room temperature. The impact strength is calculated by applying the below relationship [Dr. Fadhil A.Chyad 2011]:

$$G_c = U_c / A \quad (3)$$

Where:

G_c: is the impact strength (J/m²), U_c is the fracture energy (joule) which is determined from charpy impact test instrument.

A : cross section area of the specimen.

Fracture toughness can be calculated by using the relationship below

$$K_{Ic} = \sqrt{G_c E} \quad (4)$$

Where

K_{Ic} is the fracture toughness (MPa m^{1/2})

G_c = impact strength (J/m²)

E = modulus of elasticity (MPa) .

RESULTS AND DISCUSSION

Tensile Test

This test involves an axial tensile load being applied to standard tensile specimens to obtain the (stress–strain) curves. The ultimate tensile strength, modulus of elasticity and elongation percentage at break obtained from this test. **Fig. 4**, shows the relationship between stress and strain in tension for pure unsaturated polyester and reinforced with different weight percentage of carbon black type (N220) (0, 3, 6, 9, and 12%). It is shown from this figure that the strain increased with the increasing of stress at different rates for different composites. This character may be attributed to the increase in the flexibility of polymer chain segments by the addition of an external force [E. M. Sakr 1995].

The figure illustrates that, the stress increased with increasing weight percentage of carbon black particles until (3 wt %), then the stress decreased. That may be due to the strengthening mechanism of carbon black reinforcing filler in which, the amount of these particles plays an important role by impeding increasing the slipping of polyester resin chains. Knowing that, the chains require high stress to bend them in narrow space among the particles. The strength increases and reaches maximum values at the ratio of (3 wt. %), after which the increase of weight percentage causes the fraction between the particles and the matrix causes slipping among the particles in tension. Also the nature of the bonding between the matrix and the filler particles has an important role, i.e. the good ability of a liquid polyester to spread on the solid particles (good wettability) and may cause increasing bonding force between the matrix and the filler material, so the resultant composite will require high stress to break their physical bonding. Until the weight percentage becomes (3 wt %), the interface region between the matrix and the additive represents strengthening region. Beyond this ratio, the interface region will increase to a limit at which the discontinuity between the matrix and the filler material becomes clear and increases due to the decreasing in wettability, so that the composite material requires low stress to break their physical bonding [R. A. Abdul-Aziz 2009].

The tensile strength of composite with different filler content is given in **Fig. 5**. Pure unsaturated polyester shows tensile strength of 46 MPa. While tensile strength of composite are 54, 35, 23 and 16 MPa for 3, 6, 9 and 12 wt. %, respectively. Unsaturated polyester with 3 wt. % of nano carbon black shows maximum strength, and on further addition of nano carbon black above (3 wt. %) the tensile strength decreases. Tensile strength of nanocomposites is enhanced when the interfacial adhesion is improved. This result can be ascribed to better stress transfer at the interface between matrix and nano carbon black. The improvement of interfacial adhesion can prevent dewetting at the UPR/nano carbon black interface during tensile deformation. Therefore, well adhering nano carbon black can bear part of the load applied to the matrix and contribute to the tensile strength of the nanocomposites. For higher loading of nano carbon black in the resin matrix, nanosized-particle agglomeration is easier. Since the agglomerated particles generate defects in the material, stress concentration is likely to occur within the resin or agglomerated particles will generate slippage within the material due to external force, resulting in decreased tensile properties [Asma Yasmin 2006 and Zhang J 2003]. The decrease of tensile properties at higher weight percentage had been observed in Previous studies [Standard Methods of Evaluating the Properties of Wood-based Fibre and Particle Panel Materials 1999, Hu. Z 2001 and H.P.S. Abdul Khalil 2007]. Agglomeration and incorporation between particles and matrix were the main factor for the trend. The adsorption of the carbon filler limited wettability in the polyester matrix phase resulting in poor interface adhesion of carbon black particles to the polyester resin matrix and inefficient stress transfer between the particle matrix interface as load was applied [Ismail. H 2004].

Fig. 6 shows the relationship between the modulus of elasticity and weight percentage of carbon black. Pure unsaturated polyester shows modulus of elasticity of 942 MPa. Modulus of elasticity are 1332, 1008, 972 and 715 MPa for (3, 6, 9 and 12) wt. % nano carbon black, respectively. This figure shows that, modulus of elasticity increased with the increasing of

weight percentage of carbon black and maximum value can be achieved at (3%) weight percentage of nanocarbon black after that the modulus of elasticity will decrease with the increase of weight percentage of carbon black. This is due to the bonding force between the nanocarbon black and polymer molecules prohibited the movement of the polymer chain and reduced chain slippage. On the other hand because of the probability for the formation of a filler network is enhanced with further increase in loading which is caused by a closer distance between aggregates in the polymer system and a better filler-filler interaction [A. Mostafa 2009 and S. Chuayjuljit 2002]. The decrease of the modulus of elasticity at value more than (3wt. %) of nanocarbon black, may be attributed to the further loaded carbon black which could stiffen unsaturated polyster by replacing the polymer with rigid and non-deformable particles, which yielded higher modulus[S. Dai 2007].

Fig. 7 shows the percentage elongation at break of the composites versus the weight percentage of nano carbon black. The elongation at break (%) decreases gradually with increasing of filler loading. The reduction of elongation at break may be due to stiffening of the polymer matrix by the filler. Further increase in filler loading causes the molecular mobility to decrease due to the extensive formation of physical bonds between the filler particles and the polymer chain that stiffen the matrix [B. S. Mitchell 2004].

Hardness test

Hardness test type Shore (D) was carried out for unsaturated polyester before and after carbon black addition. **Fig. 8** shows the effect of weight percentage of carbon black on hardness. As shown hardness for the unsaturated polyester will be increased by adding carbon black and the hardness will keep increasing with increasing weight percentage, and the concept of hardness can be adopted as a measure of plastic deformation, where material will suffer under the influence of external stress and the addition of particulate fillers will contribute in raising the hardness as a consequence of increased resistance to plastic deformation [Dr.Waleed Asim Hanna 2011]. Hardness had reached its maximum value (84) at unsaturated polyester with (12 wt. %).

Impact Test

The impact test is different from the rest of the mechanical tests because it is very fast, where the specimen will be subjected to the rapid stress leading to change in the behavior of material. Results in **Fig. 9** and **Fig. 10** revealed that the impact strength and fracture toughness of unsaturated polyester increased with the increasing of weight percentage of carbon black up to (3 %wt), and reach its maximum amount and then decreased with further addition of carbon black. The presence of fine particles dispersed within the matrix makes plastic deformation easier. Therefore, during the fracture of a composite in which the nanoparticle is well dispersed, the stress will have to be bigger to start the micro crack in the UPR matrix, and the impact energy will largely be absorbed by the exhibited plastic deformation, which occurs more easily around the nanoparticles. Hence, the good carbon black dispersion resulting in less agglomeration leads to a better impact strength of the nanocomposites. When carbon black content is more than (3 wt. %), it easily agglomerates into large agglomerated particles, which will become the site of stress concentration and can act as a micro crack initiator. Therefore, a larger aggregate is a weak point that lowers the stress required for the composite to fracture and hence the impact strength of the nanocomposites would be decreased [Chi-Ming Chan 2002 and J. Spnoudakis 1984].

Bending Test

The properties of bending usually depend on the nature of the bonding between the fillers and the matrix materials. **Fig. 11** shows the relationship between the bending strength

(flexural strength) and the weight fraction of carbon black. Flexural strength of pure UPR is (40.8) MPa. Where the Flexural strength of composite are 85.4, 37, 34 and 29.7 MPa for (3, 6, 9 and 12) wt. % carbon black, respectively. The figure illustrates that the bending strength increases with increasing weight fraction of carbon black, and reaches its maximum amount at (3 wt. %). This can be related to the strengthening mechanism and the nature of the bonding between the matrix and reinforcement. The increase in bending strength may be also due to the high elastic modulus of the filler material compared with that of the matrix material [J.B. Park 2002]. Flexural strength will decrease with the increasing of carbon black. This is may be due to the agglomerations of carbon black results in inhomogeneous distribution and hence weaken the interaction between the filler and matrix. This subsequently reduces the flexural strength of carbon black composite system [R. Baskaran 2011].

Fig. 12 shows the relationship between the maximum shear stress and the weight percentage of carbon black. The maximum shear stress is obtained in (3wt. %) weight percentage carbon black. The highly (reinforcing- matrix) bonding of the composites has a remarkable effect on increasing the maximum Shear stress. The presence of carbon black particles with unsaturated polyester increases the maximum shear stress due to ability of these particles to hinder the crack propagation. Maximum Shear stress will decrease with the increasing of carbon black above (3wt. %) weight percentage. This is may be due to the agglomerations of carbon black and weaken the interaction between the filler and matrix [Israa Faisal Qhazi 2012].

CONCLUSIONS

The addition of carbon black (N220) to unsaturated polyester an improvement in the Mechanical properties. The conclusions are:

1. Tensile strength, Modulus of Elasticity, Impact strength, Fracture toughness, Flexural (Bending) strength and maximum shear stress increased with the increase of carbon black Weight percentage, maximum values were (54 MPa), (1332 MPa), (1.9 KJ/m²), (5 MPa.m^{0.5}) (85.4 MPa) and (2.06 MPa) respectively, at (3wt. %) carbon black then these properties decreased with increasing of carbon black weight percentage.
2. Elongation percentages at break decreased with the increasing of the carbon black weight percentage and maximum value achieved was (4.9%) at unsaturated polyester without addition.
3. Hardness of composites increases with the increasing of carbon black weight percentage and reaches a maximum value of (84 Shore D) for unsaturated polyester reinforced with (12 wt. %) carbon black.

Table 1 the graded of carbon black [C. A. Harper 2006].

	ASTM classification	Abbrev.	Common name	Particle size, nm	DBP absorption, ml/100 g
Furnace blacks	N110	SAF	Super abrasion furnace	21	113
	N220	ISAF	Intermediate abrasion furnace	23	115
	N326	HAF-LS	High abrasion furnace, low structure	28	72
	N330	HAF	High abrasion furnace	29	101
	N550	FEF	Fine extrusion furnace	50	120
	N660	GPF	General-purpose furnace	62	91
	N770	SRF	Semireinforcing furnace	66	75
Thermal blacks	N880	FT	Fine thermal	150	52
	N990	MT	Medium thermal	400	40
Channel blacks	S301	MPC	Medium processing channel	27	72
	S300	EPC	Easy processing channel	32	75

Table 2: Properties of Unsaturated Polyester [W.D.Callister,JR 2006].

Density (kg/m ³)	Thermal Conductivity (W/m. °C)	Tensile Strength (MPa.)	Modulus of Elasticity (GPa.)	Fracture Toughness (MPa.m ^{0.5})
1200	0.17	70.3 -103	2.06 – 4.41	0.6

Table 3: Properties of Carbon black (N220) [(ICBA) 2004].

<i>Property</i>	Ash content, %, max.	Iodine adsorption, g/kg	PH	Pour density, kg/m ³	Sulfur content, %, max.
<i>Value</i>	0.75	121 ± 4	6 - 9	355 ±20	1.5

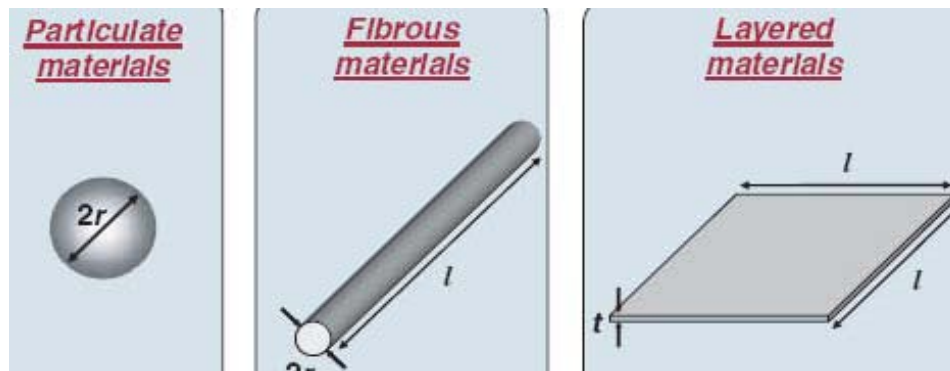


Fig. 1: Various kinds of geometries of nanoparticles[Yano. K 1993]

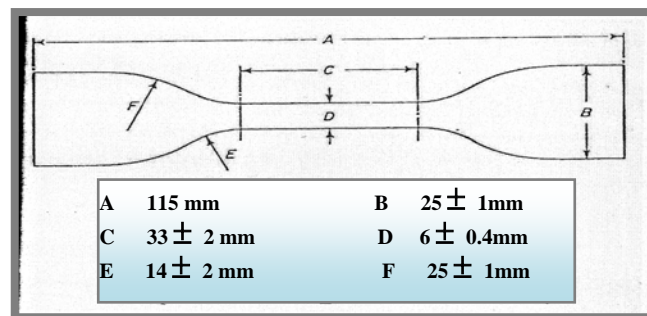


Fig. 2: Tensile Test Specimens

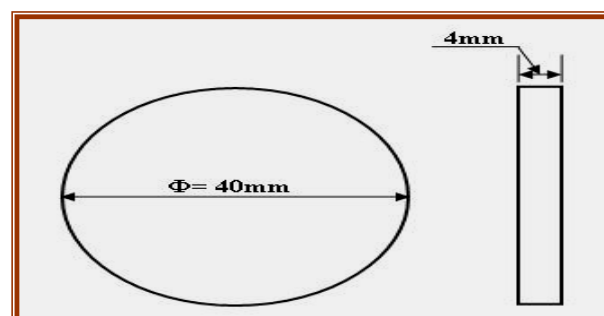


Fig. 3: Shows Schematic Specimen of Hardness

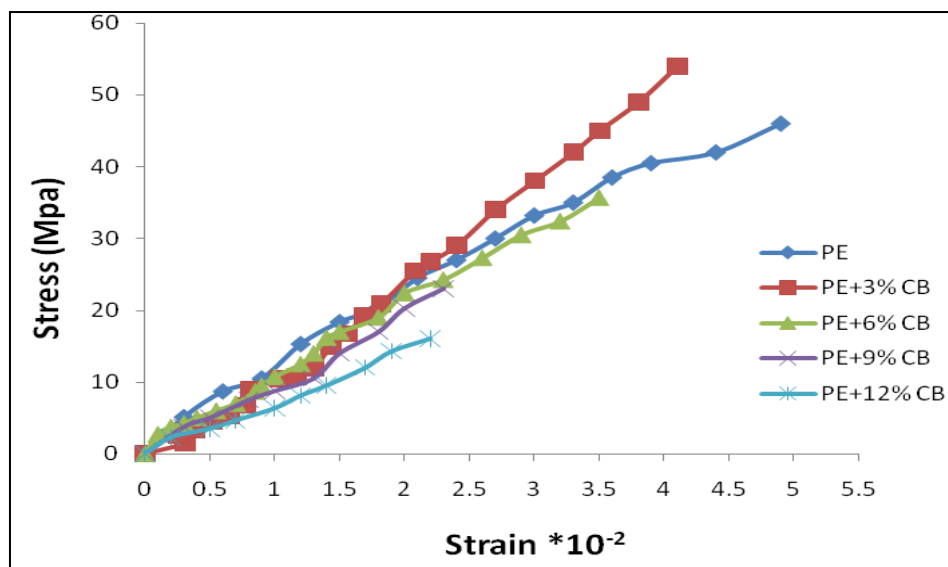


Fig. 4: Stress-Strain Curves of unsaturated polyester reinforced with Different weight fraction of Carbon Black.

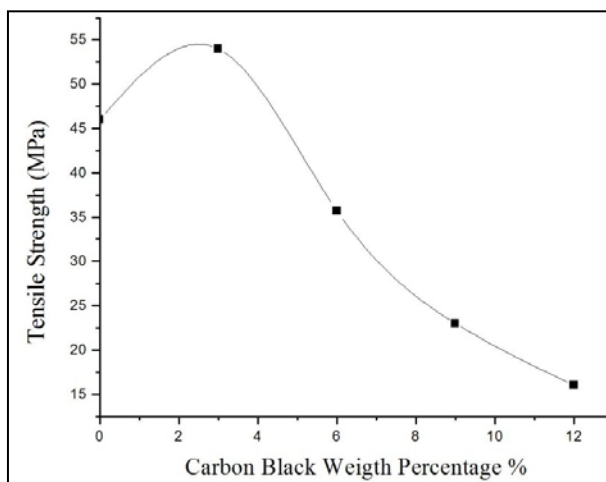


Fig. 5: Tensile Strength Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

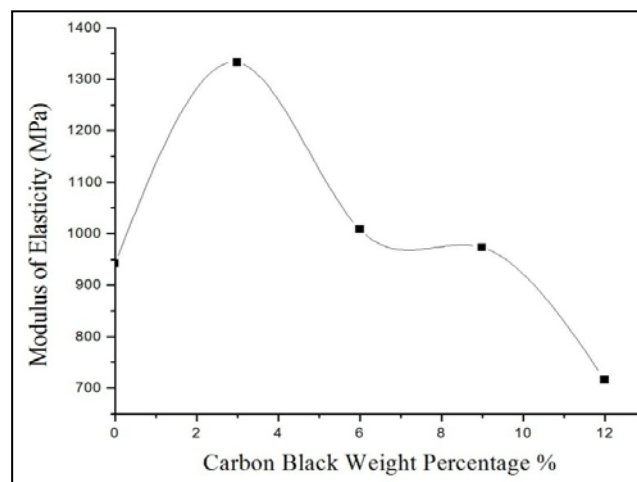


Fig. 6: Modulus of Elasticity Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

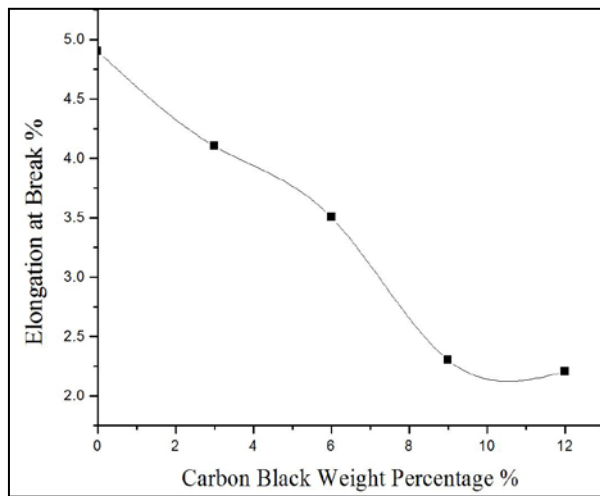


Fig. 7: Elongation at Break Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

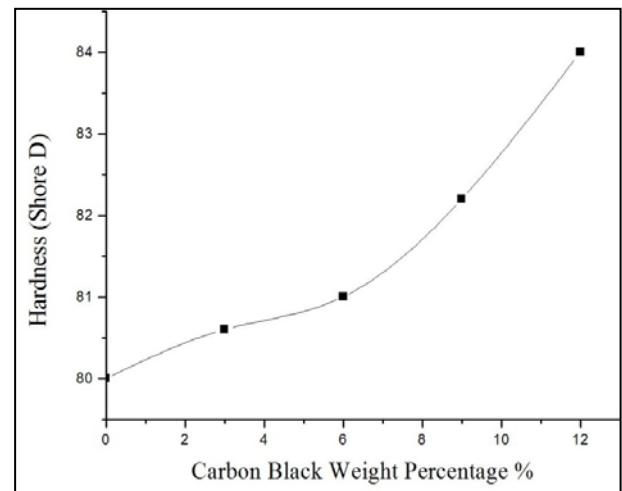


Fig. 8: Hardness Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

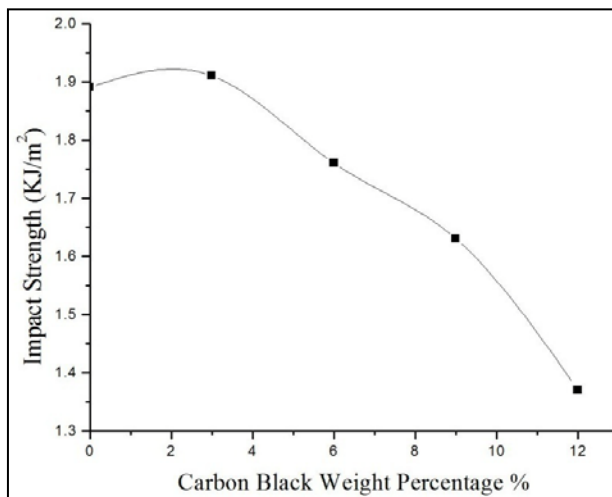


Fig. 9: Impact strength Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

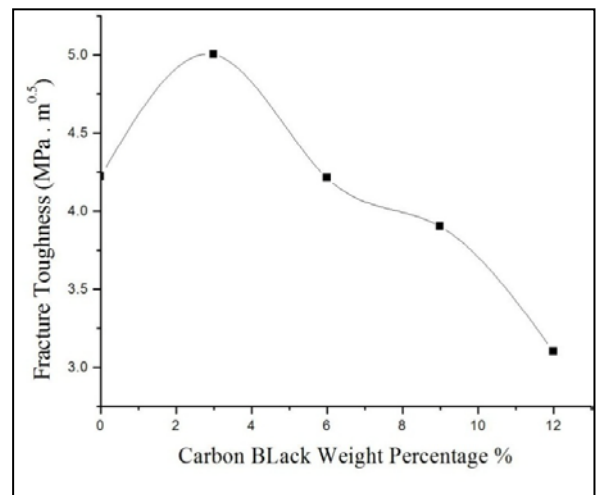


Fig. 10: Fracture Toughness Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

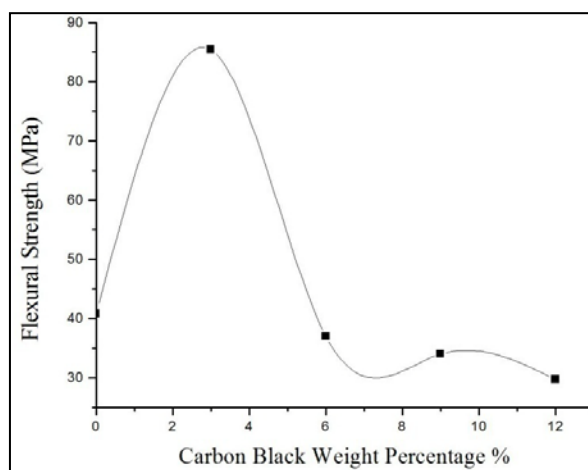


Fig. 11: Flexural strength Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

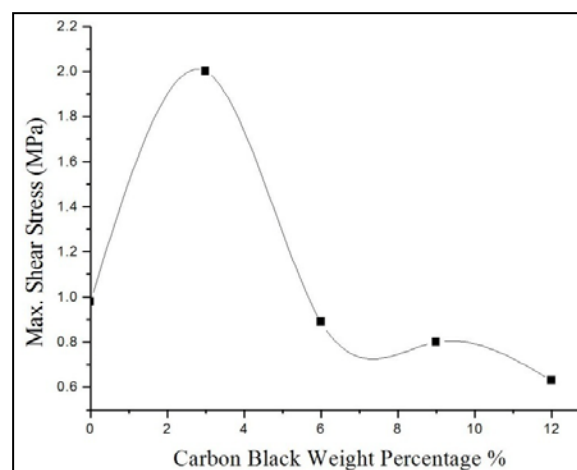


Fig. 12: maximum Shear stress Curve of unsaturated polyester reinforced with Different weight percentage of Carbon Black.

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