

THE FLEXURAL PROPERTIES OF IRAQI-BAUXITE FILLED EPOXY COMPOSITES ⁺

Thaer Abdalwahab Shihab *

Abstract:

This study is focus on the mechanical properties of Iraqi bauxite filled epoxy composites. In this study, bauxite content was varied systematically to evaluate their effect on the properties of the composites. The composites were prepared using gravity molding technique. From the flexural test results, it was observed that the flexural modulus increases with increasing bauxite content from 1.25 to 40 wt% with 40 wt% of bauxite content recorded the highest value of flexural modulus (4.93 GPa). In contrast, the flexural strength of the composites decreases with rising bauxite content till 20% and then increases with bauxite content increase from 20 to 40%. It has also been demonstrated that the pattern of flexural stress-strain curve is also affected with addition of bauxite particles. The flexural strain at break and the toughness decreases with bauxite content increase.

These types of composite materials are suitable for use in braking systems with moderate loads, but vehicle manufacturers are tending to design increasing number of vehicles with more braking power, also used in textile for heavy duty stores.

Keywords: bauxite filled composites, flexural strength, flexural modulus, flexural toughness and flexural strain at break point.

خواص الحني لمادة مركبة اساسها راتنج الايبوكسي المدعم بحبيبات البوكسيت العراقي

ثائر عبد الوهاب شهاب حمد

المستخلص :

تتمحور هذه الدراسة على الخواص الميكانيكية للمواد المركبة من الأيبوكسي المدعم بالبوكسيت العراقي. تتغير نسبة البوكسيت في هذه الدراسة بشكل منتظم لتقييم تأثيرها على خواص المواد المركبة. تم تحضير المواد المركبة باستخدام تقنية الصب. أظهرت نتائج اختبار الحني أن معامل المرونة تزداد بزيادة النسبة الوزنية للبوكسيت من 1.25% إلى 40% حيث سجلت أعلى قيمة لها عند نسبة 40% بوكسيت بقيمة مقدارها 4.93 GPa. بينما تقل مقاومة الانحناء للمواد المركبة بزيادة النسبة الوزنية للبوكسيت لغاية 20% وبعد ذلك تزداد بزيادة نسبة البوكسيت بين 20% و 40%. كما ظهر جلياً تأثير إضافة حبيبات البوكسيت على منحنيات الاجهاد-الانفعال للحني. ان انفعال الحني عند الكسر والمتانة تنخفض بزيادة محتوى البوكسيت في المادة المركبة. هذا النوع من المواد المركبة مناسب جداً في أنظمة التوقف للمركبات للأحمال المعدلة وكذلك تستخدم في أرضيات المخازن الثقيلة.

⁺ Received on 10/4/2013 , Accepted on 28/5/2014

* Assistant Lecturer / Technical College / Baghdad

1. Introduction :

Epoxy resin is widely used as a substrate material in electronic packaging industry. As one of the most widely used thermosetting resin, epoxy resin possesses special chemical characteristics such as little or no by-products or volatiles formation upon curing, low shrinkage, can be cured over a wide range of curing temperatures and control-able degree of cross-linking [1]. However cured epoxy resin also have their drawbacks that should be considered for example, as of all thermosetting resins, epoxy resin is inherently brittle with low fracture toughness, poor resistance to crack propagation and low impact strength [1].

There exist varieties of approaches to enhance the properties of epoxy resin. One of the approaches is by incorporating reinforcing fillers i.e glass-fiber [2], silica powder and aramid fiber [3] depending on the application of the products. Recently, researchers have employed nanosilica particles as filler for epoxy resin for application in electronic packaging material with improved properties of such as higher loss factor, lower glass transition temperature and higher moisture absorption [4]. Similarly, there exists variety of fillers for reinforcement of composites material. There have been a limited number of studies that provides each fillers with their reinforcement effects in each matrix system or in another words a database for engineers or scientists to choose prior to the selection of potential material as filler that suit best for certain application. Therefore, in this study was the filler based selected on the criteria to suit the structure and electronic packaging applications.

Ceramic filler, in this case is bauxite was incorporated into the epoxy resin to improve the mechanical and physical as well as thermal properties [5]. In order to maximize the reinforcement effect of bauxite, it is important to ensure that the particles are homogeneously dispersed in epoxy matrix [6]. Therefore, the optimum level of bauxite content was selected and employed to examine the flexural fractured of specimen.

Addition of red mud (an industrial waste) in to sisal fiber, banana fiber reinforced unsaturated polyester (USP) is discussed in this study. Red mud is the caustic insoluble waste residue generated during the alumina production from bauxite. Composites were fabricated separately with sisal/USP, banana/USP and each of them was filled with red mud also through compression molding process. Static mechanical tests like tensile, flexural, impact were conducted as per ASTM. Experimental results show that the addition of red mud promotes a marginal increase in the mechanical strength.[7]

The composites flexural strength and moisture resistance with modified fibers have also been investigated. It was found that chemical modification of fibers reduced the overall water uptake of the jute fibers. The flexural strength of the composite with modified fibers increases significantly compared to untreated fibers [8].

2. Experimental Work :

2.1. Materials :

Epoxy resin which form thermosetting material is combined with a hardner, which enables cross-links to establish between the epoxy molecules to produce a thermosets material. CONIPOX 77Z Epoxy type was used in the study which was produced by Conica Techik AG and consists of two components of a high grade,

low viscosity, solvent free, colorless material, the density at 20°C is approximately 1.09 g/cm³ and has a mixing ratio of 100:45 based on weight, or 2:1 based volume, application time of 30 min at approximately 20°C and after solidification process, and it demonstrates low density 1.33 gm/cm³, and high electrical resistance. The reaction product is bi3-phenal A (Epichlorhydrin) with average molecular weight \leq 700. CONIPOX 77Z meets the requirements set down by ZTV-TP-EP of German Federal Transport Ministry-System Test of ZTV-BEL-B-3187 and BBA No.

The Iraqi bauxite deposit was discovered in north Wadi Hussainyat in the Western Desert of Iraq has a density of 3.08 g/cm³ and an average particle size of \leq 36 μ m. The chemical analysis of bauxite are listed in Table (1).

Table (1): Chemical Analysis of Iraqi Bauxite According to General Foundation of Geological Survey.

Oxide	Al ₂ O ₃	SiO ₂	FeO ₃	Na ₂ O	K ₂ O	SO ₃	Moisture	L.O.I
%	65.6	14.62	1.43	0.17	0.53	0.45	0.24	1.29

2.2. Sample preparation :

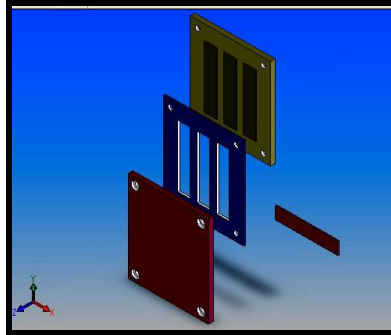
Prior to mixing, the bauxite powder was dried in an oven at 100°C for 1 hour. The mixture of bauxite and epoxy was then mixed in a container with continuous stirring at 200 rpm for 15 minutes and 100 rpm for 5 minutes. Hardener (50% by volume from epoxy resin) was added at 6 minutes of stirring and the mixture was further stirred for another 6 minutes. The mixture was degassed in the tube vacuum oven for 30 minutes and then was quickly poured into steel mold shown in figure (1) which is covered with the silicone mold release agent and wax film. The mold temperature was kept at 60°C and subjected to uniform pressure for another one hour.

2.3. Flexural Testing :

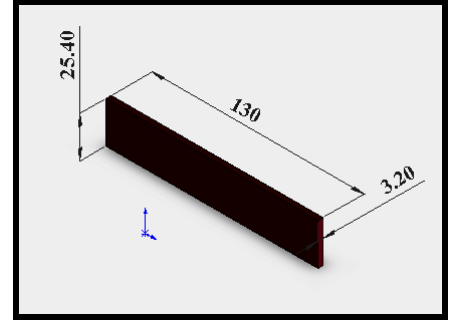
Flexural tests under three-point bend configuration were performed according to ASTM D790-M86 and confirmed to the dimensions in Figure (1) with a length to depth ratio of 32:1, 130mm length, 100mm support span, with 5 mm diameter for nose and two supports.



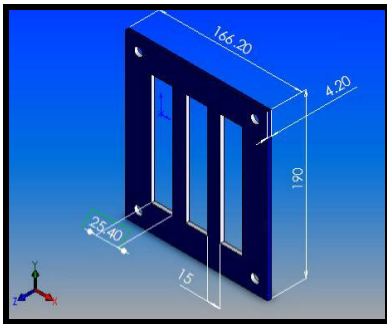
Photo of Steel Die Cavity



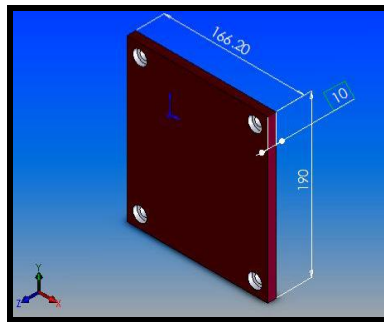
Disassembly of Die Parts



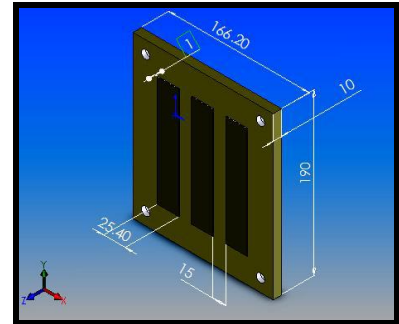
Standard Specimen Dimensions
According to ASTM D 790 - M86



Die Cavities



Lower Plate



Punch (Upper Plate)
All Dimensions in Millimeters

Figure (1): Photos & Design Drawings of Flexural Specimen Mold Die

The machine was run under displacement control mode at a cross head speed of 1.5 mm/ min and all the tests were performed at room temperature.

The values of flexural strain and flexural modulus were calculated based on Equation (1) and (2), respectively.

$$\varepsilon = \frac{6 D d}{L^2} \dots \dots \dots (1)$$

where:

- ε = strain in the outer surface, (mm)
- D = maximum deflection at the center of the beam, (mm)
- L = support span, (mm) and
- d = depth, (mm)

$$E = \frac{L^3.m}{4 b d^3} \dots \dots \dots (2)$$

where:

- E = modulus of elasticity in bending, (MPa)
- L = support span, (mm)

- b** = width of beam tested, (mm)
d = depth of beam tested, (mm)
m = slope of the tangent to the initial straight-line portion of the load-deflection curve
 N/mm² of deflection.

3. Result and Discussion :

3.1. Processing of Composites :

The optimum level content of bauxite content (by weight percent) was dictated by the process-ability of the mixture between bauxite particles and epoxy resin, typically the viscosity of the mixture. It is clear that the mixture become too viscous when the bauxite content approaching 45 wt% of epoxy. Therefore, the optimum bauxite content in this study was kept to a maximum of 40 wt% of bauxite. This is important to ensure that the bauxite particles are readily and easily dispersed in the epoxy matrix.

3.2. Flexural Stress – Strain curves :

Flexural properties are strongly affected by the quality of the interface in composites, i.e. the static adhesion strength as well as the interfacial stiffness, which plays a major role in promoting the filler reinforcement [9]. It has been proven that the flexural stress-strain could be used to study the changes induced by addition of filler. In this study, we have analyzed the changes in terms of flexural stress-strain curves with addition of bauxite into the epoxy resin.

Figure (2) shows the flexural stress-strain curves recorded at 1.5 mm/min of strain rate for various filled and unfilled systems. In general, stress-strain curve provides useful information pertaining to the flexural properties such as flexural modulus, flexural strain and flexural strength of the composites [10]. From Figure 1, it is clear that the slope of stress-strain curves increase with increasing bauxite content (20 wt% - 40 wt %). Theoretically, the slope of the flexural stress-strain curve is directly proportionate to the flexural modulus of the composite which associated with flexural rigidity and bending stiffness of the composite beam. The increase is expected since the bauxite particle is inherently rigid and thus influences the rigidity of the composite as a whole (bulk). This indicates that the composite becoming increasingly brittle with lesser deflection is observed during flexural loading. However, it is also clear that the addition of 1.25 wt% to 20 wt% of bauxite slightly reduces the level of stress at yield of the composites. From the same figure, it can also be seen that the degree of plastic deformation reduces with increasing bauxite content. Ability of the material to plasticity deformed is largely determined by the mobility of the molecular chain (molecular motion) to take place under applied load. The presence of rigid particles such as bauxite in this case has restricted the mobility of the molecular chain to pass each other and orientation which consequently resulted in instantaneous failure (brittle failure) area before the yield stress is reached. However, the benefit that one material can gain in the presence of the rigid

particle is the increase in rigidity (stiffness) that is indicated by an increase in the steepness of the stress-strain curve in the elastic region.

All stress-strain curves are subjected to the same mathematical Hoerl model with high correlation coefficients and best standard deviations as illustrated in Table (2) which is approved by Namer [11] for the PMC's and their hybrids for the same CONIPOX 77Z Epoxy type.

The data of stress-strain curves are input to Curve-Expert 3.1 software and through the selection of best curve finder state that the Hoerl model is the first selections.

Table (2): Mathematical Hoerl Model Constants for Stress-Strain Curves of Bauxite Filled Epoxy composites

Bauxite (wt%)	Hoerl Model Constant $y = a(b^x).(x^c)$			Standard Deviation	Correlation Coefficient
	a	b	c		
0	36.598	0.710246	1.59359	0.304438	0.99993857
1.25	37.20887	0.750612	1.38345	0.299201	0.99994072
2.5	36.40219	0.747673	1.416682	0.214291	0.99997187
5	38.19962	0.765296	1.311402	0.114806	0.99999047
10	39.45259	0.765327	1.284666	0.297697	0.99994348
20	42.34687	0.712758	1.416473	0.222704	0.99996559
40	72.1828	0.906315	0.699124	0.128303	0.99999221

In general, these Horel Model methods are applicable in any experiment involving multiple quantitative treatment factors. Specifically these methods should be used first, where biologically relevant interactions among treatments are expected and second, where drawing accurate conclusions or making appropriate recommendations depends on the ability to accurately characterize these interactions.[12]

The design combined a four factors face-balanced cube with a 2^4 factorial to produce a design with five levels per treatment and 41 treatment combinations observed. Alternative designs can be constructed using either statistical optimal design theory or computer-assisted design (e.g., using that the SAS PROC OPTEX). Finally, while we have shown that the Hoerl is a useful general purpose model for exploratory plant nutrition research, it is not the only model available [12].

3.3. Flexural Strength :

Flexural strength of a material is defined as the ability to resist deformation under load [9]. From Figure (3), flexural strength is decreased with the increasing of bauxite weight fractions up to 20wt%. The increase in flexural strength is very unlikely with farther addition of bauxite particles due to the incompatibility between bauxite and epoxy. Weak interfacial adhesion between bauxite and epoxy is the major contributor to the deterioration of flexural strength of the composite. In contrast, at 40 wt% bauxite, the value of flexural strength is slightly extreme

affected with addition of bauxite as compared to 20 wt%. with compared this study with Mohamed study (2011) [13] that used the same type of matrix conipox 77Z but with copper particles as a reinforcement shows that the flexural strength reduced as weight fraction of reinforcement till 15%.

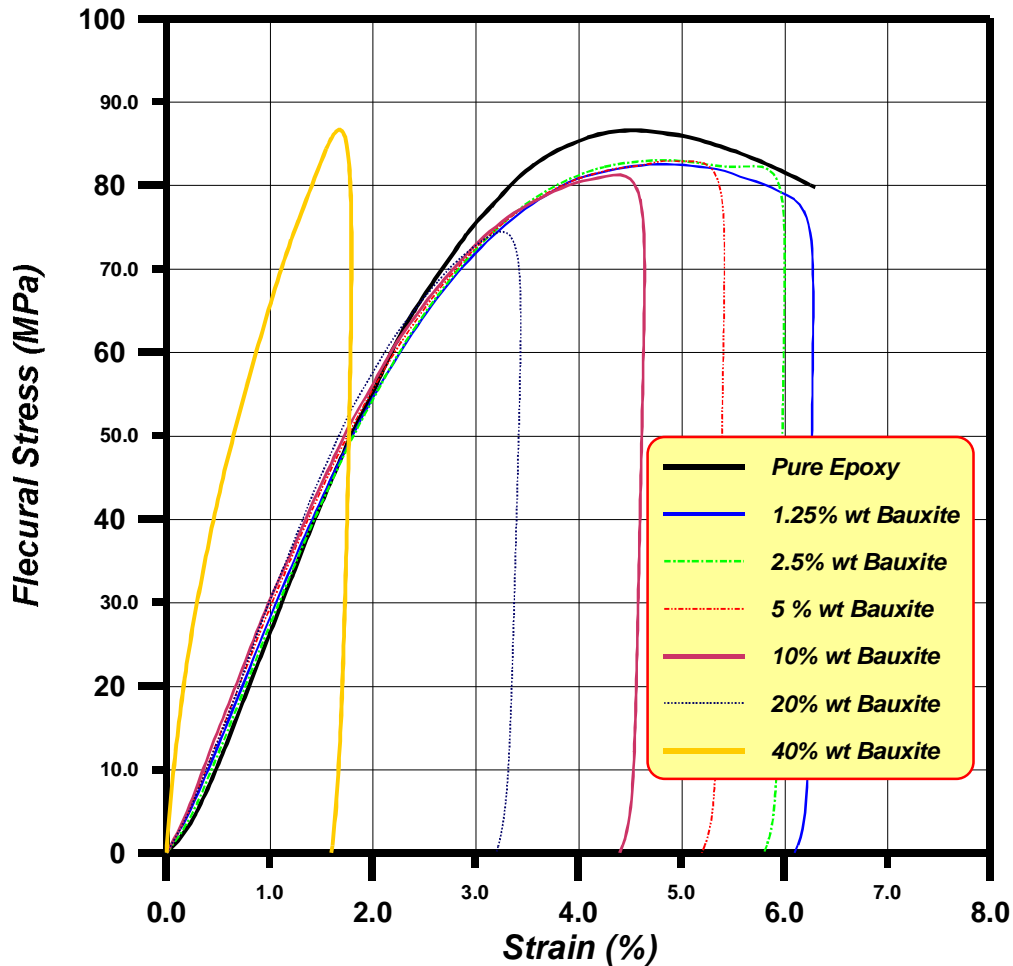


Figure (2): The Stress-Strain Curves of Bauxite Filled Epoxy Composites.

3.4. Flexural Break Strain :

As stated before, the strain at break usually declines with the addition of filler content. The declining trend usually continues with increasing filler content. Similar trend is also observed in this study. Significant drop in the value of strain at break was observed at filler loading as low as 1.25wt%, (see Figure 3). In polymer matrix composites (PMC), most of the deformation occurs in the matrix phase. As the percentage of bauxite weight fraction increases, the ability of the matrix phase to deform plasticity is also reduced as shown in Figure (4) due to the reason as explained previously. Rigidity of bauxite particles has also directly responsible for the decreases in the flexural strain at break value.

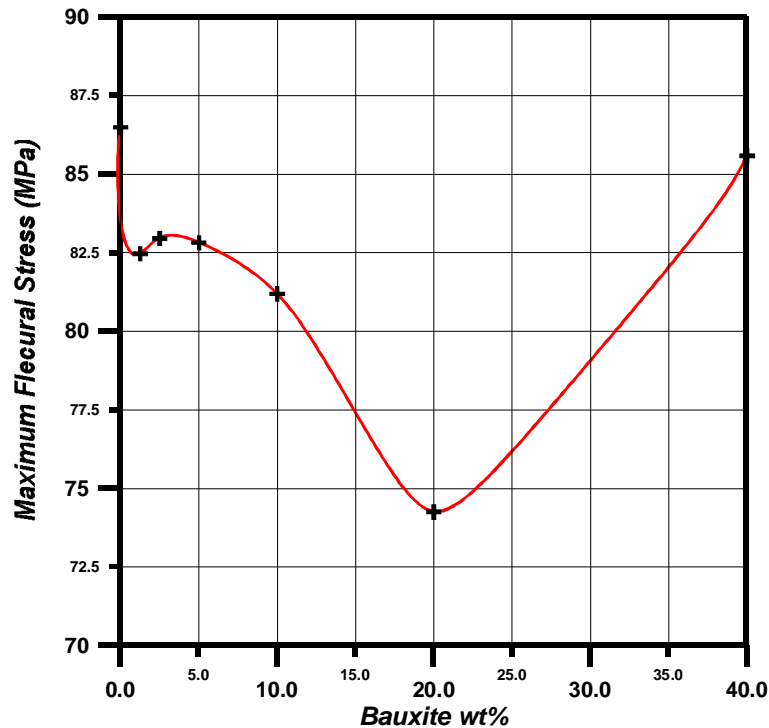


Figure (3): Flexural Strength of Epoxy Composites as a Function of Bauxite Weight Fractions

3.5. Flexural Modulus :

Flexural modulus is the ratio of stress to strain within the elastic limit (when measured in flexural mode) and this property was used to indicate the bending stiffness of the material [8].

Figure (5) shows the effect of bauxite weight fractions to the flexural modulus of the composites. As expected, the flexural modulus of the composite increases with the addition of bauxite content. It is widely accepted that the addition of the rigid filler will increase the modulus of the composites materials following the Rule of Mixtures [9]. A drastic increase in flexural modulus of the composites is observed at 40wt% of bauxite content with approximately an increase of 53% from the 20wt% of bauxite and 68% of the neat epoxy. The increasing in flexural modulus is mainly attributed to the inherent stiffness of bauxite particles and the restriction of chain mobility as explained previously.

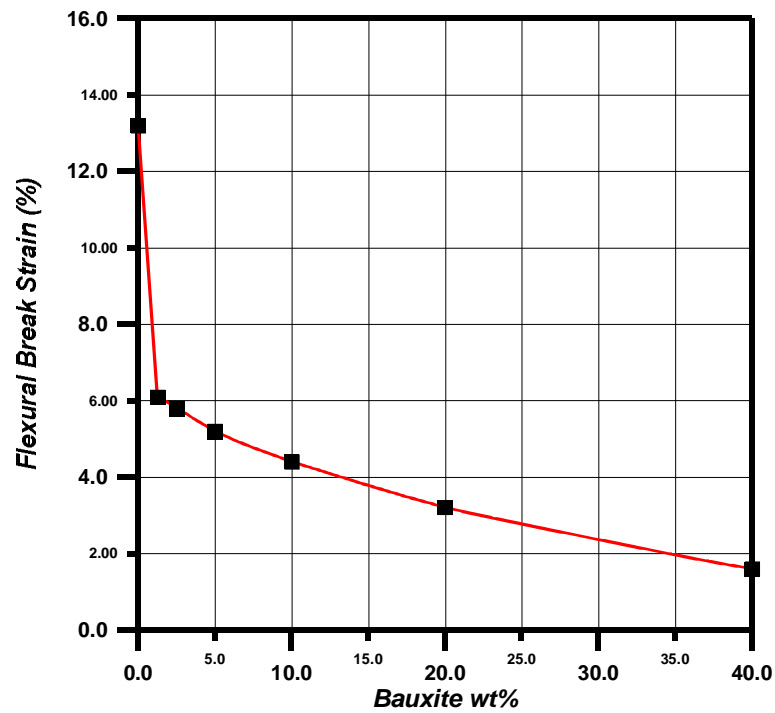


Figure (4): Flexural Break Strain of Epoxy Composites as a Function of Bauxite Weight Fractions.

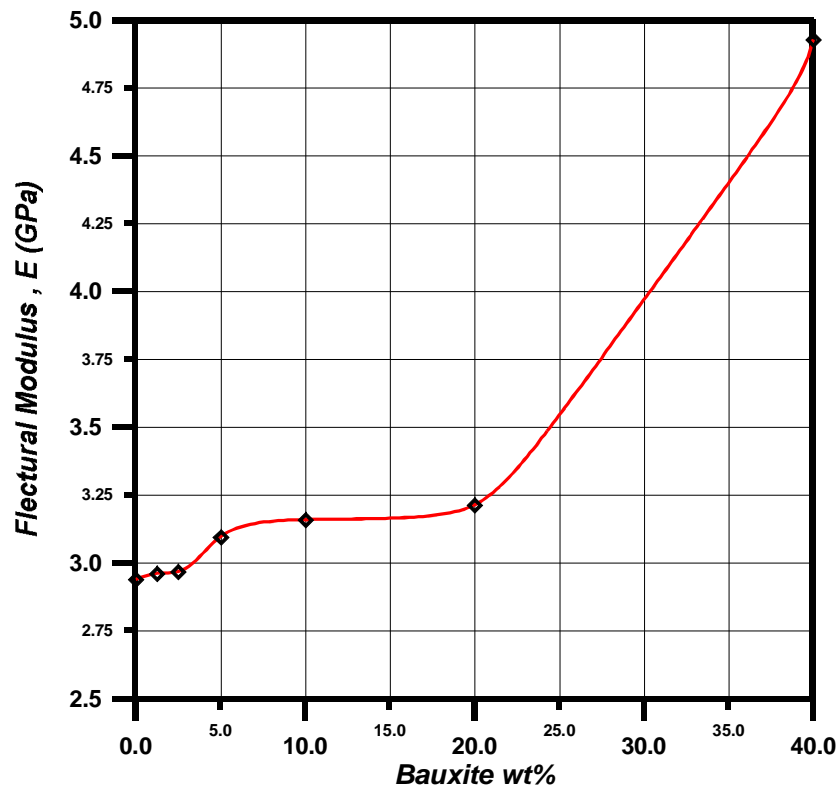


Figure (5): Effect of Bauxite wt% on Flexural Modulus of Composites

3.6. Toughness :

The area under the stress-strain curve can be used to calculate the deformation energy or sometimes known as toughness of the materials. In this study area under each of the stress-strain curve is calculated by using the proficiently curve expert software. Summary of the toughness corresponding to the area under stress-strain curve as a function of bauxite weight fractions is presented in Figure (6). It can be observed that, the toughness of the bauxite filled epoxy composites was reduced with the increase in the bauxite wt% content. In theory, the toughening mechanism in composites is affected by several factors such as size, morphology and volume fraction of the reinforcement, interfacial bond and the properties of the matrix and also the filler phase transformations [9].

Figure (6) clearly shows that the addition of bauxite wt% content has drastically reduced the interfacial adhesion between matrix and filler. Therefore, when the external load is applied to the specimen the weak adhesion between bauxite and epoxy could not sustain the load. In comparison, the toughness of pure epoxy is 3.95 N.m as compared to 0.86 N.m for 40 wt% bauxite content. The huge drop is attributed to the poor adhesion and restriction of chain mobility in the epoxy matrix.

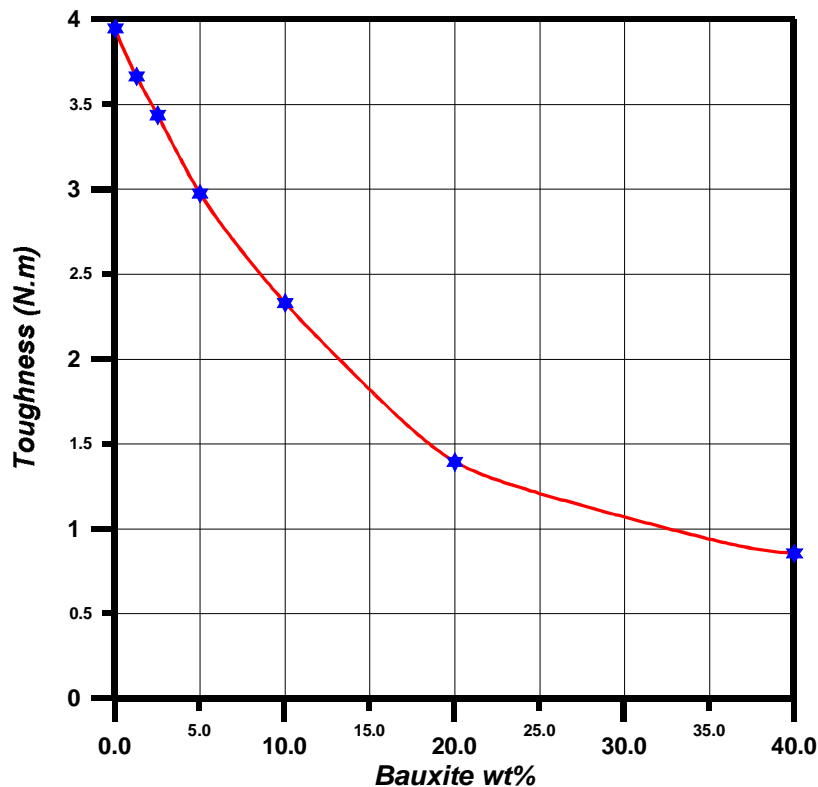


Figure (6): Effect of Bauxite wt% on Composites Toughness.

4. Conclusion :

The bauxite reinforced epoxy composites with various bauxite weight fractions have been successfully produced. Optimization study on the composition of bauxite in the composites reveals that **40 wt%** of bauxite of $\leq 36\mu\text{m}$ size and irregular shape gives best overall mechanical performances. Beyond 40 wt% the mixture of epoxy with α -alumina is hardly processable under normal processing conditions. Calculation on the deformation energy indicated that the toughness reduced with increasing bauxite loading in the composites associated with reduced interfacial bond between bauxite and matrix. Mathematical Hoerl Model represents the stress strain curves with high correlations and best standard deviations and showed good agreement with the experiment data over the range of bauxite content studied.

5. Acknowledgement :

The author is grateful to the ministry of Science & Technology / Materials research center and General Foundation of Geology Survey for providing the financial assistance that resulted in this paper. Also to Asst. Prof. Dr. Nasri S.M. Namer for his assistance by die and specimen preparation equipment.

6. References :

1. Hodgkin, J.H., Simon, G. P., and Valery, R. J., "*Thermoplastic Toughening of Epoxy Resins: a Critical Review*", Polymers for Advanced Technologies, Volume 9, Issue 1, pages 3–10, J.Wiley online library, January 1998.
2. Chakrapa Tuakta, "*Use of Fiber Reinforced Polymer Composites in Bridge Structure*", Master of Engineering in Civil and Environmental Engineering at the Massachusetts Institute of Technology, USA, June 2005.
3. Akbar Afaghi-Khatibia, and Yiu-Wing Maib,c,"*Characterisation of fibre/matrix interfacial degradation under cyclic fatigue loading using dynamic mechanical analysis*", Composites: Part A 33, 1585–1592, Elsevier Science Ltd. All rights reserved, 2002.
4. Yangyang Sun, Zhuqing Zhang, and C. P. Wong,"*Influence of interphase and moisture on the dielectric spectroscopy of epoxy/silica composites* " Journal: Polymer , vol. 46, no. 7, pp. 2297-2305, 2005.
5. Erfan Suryani Abdul Rashid, Hazizan Md Akil, Kamarshah Ariffin, and Chee Choong Kooi "*The Flexural and Morphological Properties of α -Alumina Filled Epoxy Composites*", Malaysian Polymer Journal (MPJ), Vol 1, No. 1, p 25-38, (2006).
6. Hussain M.; Oku Y.; Nakahira A.; and Niihara K, "*Effects of wet ball-milling on particle dispersion and mechanical properties of particulate epoxy composites*", Materials Letters, Volume 26, Number 3, pp. 177-184(8), (1996).
7. V. Arumuga Prabu, V. Manikandan, M. Uthayakumar, S. Kalirasu, "*INVESTIGATIONS ON THE MECHANICAL PROPERTIES OF RED MUD FILLED SISAL AND BANANA FIBER REINFORCED POLYESTER COMPOSITES*", Materials Physics and Mechanics 15, 173-179, (2012).
8. B. C. Patel, S. K. Acharya, D. Mishra, "Environmental effect of water absorption and flexural strength of red mud filled jute fiber/polymer composite", International Journal of Engineering, Science and Technology, Vol. 4, No. 4, pp. 49-59, 2012.
9. Wetzel B, Hauptert F, Zhang MQ. *Epoxy nanocomposites with high mechanical and tribological performance. Journal of Composites Science and Technology*: 63: 2055-2067, (2003).
10. Matthews. F.L., Rawlings. R.D. *Composite Materials: Engineering and Science*, United Kingdom: Chapman & Hall, (1994).
11. Selvin,T.P., Kuruvilla, J., Sabu, Thomas. *Mechanical properties of titanium dioxide-filled polyester microcomposites*, Materials Letters, 58: 281-289, (2004).
12. Namer, Nasri. S.M., "*Mechanical Behavior of PMC's and their Hybrids Reinforced with Copper, Carbon and Graphite Particles*", Ph.D. Thesis, Machines & Tools Engineering Department, University of Technology, 2007.
13. Mohammed Abdulsattar Mohammed , "*Mechanical Behavior for Polymer Matrix Composite Reinforced By Copper Powder*, Nahrain University, College of engineering Journal (NUCEJ) Vol.14 No.2, pp.160-176, 2011.
14. Ellen T. Paparozzi, Walter W. Stroup, and M. Eliizabeth Conley, "*How to Investigate Four-way Nutrient Interactions in Plants: A New Look at Response Surface Methods.*", J. Amer. Soc. Hort. Sci. 130(3): 459-468. 2005.