Study of Mechanical Properties of Epoxy / Rice Husk Filler Composites

Ahmed F.Hamza
University of Babylon – College of Materials Engineering

Summery

This paper study the tensile and flexural properties of composites materials made from rice husk as filler particles and epoxy resin as matrix . The tensile and flexural tests of composites based on rice husk filler particles at three different filler contents 5 wt.%, 10 wt.%, and 15 wt.% , were carried out using universal testing machine according to ASTM D638 and ASTM D790 respectively. Experimental results showed that tensile and flexural properties of the composites constraint increased with the increase of the filler particle content. The composite materials nearly linear behavior and sharp fracture for tensile and slight non-linear behavior and sharp fracture for flexural testing. The relation between stress and content of filler for tensile and flexural tests were found to be linear . Concerning the relation between the modulus and percentage of filler for tensile and flexural tests, it was found to be a quadratic relation. The same behavior was observed for the strain versus percentage of filler for tensile and flexural tests.

Key words: epoxy resin ; polymer matrix ; rice husk; composite materials ; tensile properties ; flexural properties.

دراسة الخواص الميكانيكية للمادة المركبة (ايبوكسي – قشرة الرز)

احمد فاضل حمزة

جامعة بابل / كلية هندسة المواد

الخلاصة

البحث الحالي تضمن دراسة خواص الشد والانحناء للمادة المركبة المتكونة من حبيبات قشور الرز كمادة مالئة وبوليمر الايبوكسي كمادة اساس. تم اجراء اختبار الشد والانحناء للمادة المركبة بثلاث تراكيز من المادة المالئة هي (5% ,10% , 15%) كنسبة وزنية . بينت النتائج العملية ان خواص الشد والانحناء للمادة المركبة قيد الدراسة تزداد مع زيادة محتوى المادة المالئة وان سلوك المادة المركبة يكون خطيا مع كسر حاد في اختبار الشد في حين ينحرف قليلا عن السلوك الخطي مع كسر حاد في اختبار الانحناء ،كما بينت النتائج ان العلاقة بين الاجهاد ومحتوى المادة المالئة لكلا الاختبارين هي علاقة خطية , اما العلاقة بين معامل المرونة والنسبة المئوية للمادة المالئة هي من الدرجة الثانية كما ان نفس هذا السلوك قد وجد للعلاقة بين الانفعال والنسبة المئوية للمادة المالئة في اختباري الشد والانحناء .

Introduction

Epoxy resins are one of the most important classes of thermosetting polymers which are widely used as matrix for fiber-reinforced composite materials and as structural adhesives [Zhikai *et al*. 2002, Shangjin *et al*. 2004, May and Tanaka 1983]. They are amorphous, highly cross-linked polymers and this structure results in these materials possessing various desirable properties such as high tensile strength and modulus, uncomplicated processing, good thermal and chemical resistance, and dimensional stability [Zhikai *et al.* 2002]. However, it also leads to low toughness and poor crack resistance, which should be upgraded before they can be considered for many end-use applications [Zhikai *et al.* 2002, Shangjin *et al*. 2004]. One of the most successful methods of improving the toughness of epoxy resin is to incorporate a second phase of dispersed rubbery particles into the cross-linked polymer [Drake *et al*. 1992, Yorkitis 1994]. Because the addition of rubbery materials to epoxy resins has been shown to lower their glass transition temperature (Tg) and thermal and oxidative stability, high performance thermoplastics have been employed to toughen epoxy resins in recent years [Zhikai *et al*. 2002, Shangjin *et al.*2004].

Using natural fillers to reinforce the composite materials exhibits the following interests in comparison with mineral fillers [Herrara-Franco *et al*. 2002]:

1. strong and rigid
2. light weight
3. environmental friendly
4. economical
5. renewable and abundant resource

On the other hand, the disadvantages of the materials are summarized below [Maulida *et al.* 2000]:

1. degradation by moisture
2. poor surface adhesion to hydrophobic polymers
3. non-uniform filler sizes
4. not suitable for high temperature application
5. susceptibility to fungal and insect attack

Various works on the application of natural fillers and fibers in composites like pineapple husk , sisal, coconut coir, jute, palm, cotton, bamboo, and wood as the reinforcements in composites have been reported in the literature. [Luo and Netravali 1999] studied the tensile and flexural properties of the green composites with different pineapple husk fiber content and compared with the pure resin. Sisal fiber is fairly coarse and inflexible. It has good strength, durability, ability to stretch, affinity for certain dyestuffs, and resistance to deterioration in seawater. Sisal ropes and twines are widely used for marine, agricultural, shipping, and general industrial use[Herrara-Franco *et al*. 2002]. [Cazaurang *et al.* 2001] carried out a systematic study on the properties of henequen fiber and pointed out that these fibers have mechanical properties that are suitable for reinforcing thermoplastic resins. [Ahmed *et al*. 2003]carried out research work on filament wound cotton fiber reinforced for reinforcing high density polyethylene (HDPE) resin , and they found the mechanical properties increase with increase the volume fraction of fibers. [Khalid *et al.* 2003] also studied the use of cotton fiber reinforced epoxy composites along with glass fiber reinforced polymers , and they noted that use of cotton fibers improving resistance of crushing. [Fuad *et al.* 2004]investigated the new type wood-based filler derived from oil palm wood flour (OPWF) for bio-based thermoplastics composites by thermogravimetric analysis and the results are very promising. [Schneider and

Karmaker 1996] developed composites using jute and kenaf fiber and polypropylene resins and they reported that jute fiber provides better mechanical properties than kenaf fiber.

Composites of high strength can be used in the broad range of applications as, building materials, marine cordage, fishnets, furniture, and other household appliances[Zhikai *et al*. 2002]. The objective of this paper is to study the tensile and flexural properties of epoxy composites based on rice husk filler particles.

Experimental

1- Materials

a- Polymer matrix :

Polymer system based on epoxy resin was used as matrix for the polymer composition. The resin used provided by (S.I.R) company/Saudi Arabia , and have the physical properties shown in table (1) .

b- Rice husk filler :

The rice husk was used as a filler of the epoxy resin , the husk separated from rice then washing and dried in electrically oven at (90 ºC) for (4) hours to remove absorbed moister before to it use in the polymer composite . The addition concentration of rice husk as filler was determined not exceed (15 wt.%) **.**

Filler particles have been mechanically milled in an electrical miller in dry condition at room temperature for (45) minutes . After milling the rice husk was fractioned and fraction (250 µm) by electrical sieve of the grains was used in the composite material .

2- Specimens :

Standard specimens were used in mechanical tests which made an experimented to evaluate the role of filler in composite properties . The specimen was fabricated at room temperature using hand lay-up method. The preparation of specimen includes the rice husk was added into polymer matrix and mixed the mixture by electrical mixer under mixing condition defined as rotation speed of the mixer (300 rpm) and time of mixing (6 minutes) until homogeneous mixture was obtained .

The hexamethylenetetramine (HMTA) were used as hardener, the concentration of hardener was (20 wt.%) of the epoxy weight and they were added consecutively to the mixture for (10-12 sec) for each addition. The specimens were cast in steel moulds of a size specified in the standard , before the mixture was placed inside the mould, the mold has initially been polished with a release agent to prevent the composites from sticking onto the mold upon removal . Finally the mixture was poured into the mold and left at room temperature for 24 hours until the mixture was hardened , it was removed from the mold and placed inside an oven and cured at (80ºC) for 4 hr .

3- **The strength characteristics measurements :**

a- Tensile test :

Tensile tests indicate the yield (breaking) strength of a given specimen under a tensile load , tensile strength is measured in (MPa) the shape and dimensions of specimens to be based on property (ASTM D638) [ASTM 2002].Tensile strength indicates the ability of a composite material to withstand forces that pull it apart as well as the capability of the material to stretch prior to failure.. Tensile tests were carried out using an universal testing machine, with maximum load of 30 kN.

b- Flexural test:

Flexural strength is the ability of the composite material to withstand bending forces applied perpendicular to its longitudinal axis ,flexural strength is measured in (MPa) and was determined by three point bending method and test device at a uniform loading speed flexural tests were carried out using an universal testing machine, with maximum load of 30 kN according to (ASTM D790) [ASTM 2007].

Three specimens per test condition should be carried out for testing the polymer matrix composite materials and all tests were carried out according to the above mentioned standard. Three different filler contents by weight percentage were used namely 5%, 10%, and 15%, as the maximum amount of fillers that can be added to the resin is limited to 15 % by weight only as beyond that the epoxy resin cannot accommodate the fillers, because when we add fillers more than 15% was not obtained homogeneous mixture.

Result and Discussion

a- Tensile test :

Mechanical properties of the natural filler composites depend on several factors such as the stress–strain behaviors of filler and matrix phases, the phase volume fractions, the filler concentration, the distribution and orientation of then fillers relative to one another[Herrara-Franco *et al*. 2002] .**Fig. (1)** shows the tensile stress against strain curves for rice husk filler composite. It can be seen that tensile strength and elastic modulus of the composites increase with an increase of the filler content. The composites materials nearly linear behavior and sharp fracture.

The ultimate engineering stress, in tension, that may be sustained without fracture in known as maximum tensile stress. The change in length of a specimen (in the direction of applied stress before fracture) divided by its original length in known to maximum tensile strain. The effect of rice husk filler content on maximum tensile stress, strain, and modulus of elasticity are shown in **Fig. (2–4)** respectively. The increase of the filler content, results in the increase in tensile stress and tensile modulus of elasticity. This is due to the fact that rice husk filler particles strengthen the interface of resin matrix and filler materials. The maximum tensile strength for 15% filler composite was higher (35.4 MPa) compared to other two concentration. Analytically, the relation between tensile stress and concentration of filler is found to be in linear form, as shown in equation (1**)**, in case of modulus of elasticity, the relation with the concentration of rice husk is quadratic as shown in equation (2**)**

$$\sigma_t = 0.73 w_f + 24.41 \qquad \ldots\ldots\ldots\ldots\ldots\ldots \; (1)$$

$$E_t = -0.0307 w_f^2 + 0.8225 w_f - 1.005 \qquad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where :

σ_t is the tensile stress (MPa)

E_t is the tensile modulus of elasticity (GPa)

W_f is the weight percentage of filler content

However, the tensile strain decreases following a quadratic form with an increase of the filler content due to the fact that the composite becomes harder with the increase in filler content. Therefore, the elongation of materials decreases as the fillers reduce the ductility of matrix. The relation between tensile strain and concentration of filler as shown in equation (3**)**

$$\varepsilon_t = -0.00006 w_f^2 + 0.00079 w_f + 0.0145 \qquad \ldots\ldots\ldots\ldots\ldots (3)$$

Where :

ε_t is the tensile strain (mm/mm)

Analytically results (obtained from experimental results) agreement with other researchers [Zhikai *et al*. 2002, Herrara-Franco *et al*. 2002].

b- Flexural test:

Stress at fracture from a bend or flexure test is known as flexural stress. **Fig. (5)** shows the typical flexural stress against strain curves for three different epoxy/ rice husk filler composite materials. The effect of rice husk filler content on maximum flexural stress, strain and modulus of elasticity are shown in **Fig. (6–8)** respectively. Almost similar pattern were obtained from the flexural mode as was obtained from tensile test. The maximum flexural strength for 15% filler composite was higher (77.3 MPa) and other two (5% and 10%) concentration exhibited lower flexural strength. At lower concentration of the filler material, specimen slight non-linear behavior and sharp fracture. This means that specimen deformed plastically immediate after elastic deformation.

The increase of filler content results in the steady linear increase in flexural stress and quadratic increase of flexural modulus of elasticity, which can be seen in equations (4) and (5) respectively.

$$\sigma_f = 2.3 w_f + 43.0333 \qquad \ldots\ldots\ldots\ldots\ldots.(4)$$

$$E_f = -0.07904 w_f^2 + 2.1684 w_f - 6.855 \qquad \ldots\ldots\ldots\ldots\ldots.(5)$$

Where :

σ_f is the flexural stress (MPa)

E_f is the flexural modulus of elasticity (GPa)

W_f is the weight percentage of filler content

This increase is due to the relationship between the interface of fillers and matrix in which the fillers strengthen the composite materials. However, the strain decreases following a quadratic form with the increase in the filler content due to the fact that the materials have become harder with the increase in filler content, as obtained for tensile testing. Therefore, the elongation decreases as filler materials reduce the ductility of matrix. The relation between strain and concentration of filler as shown in equation (6**)** .

$$\varepsilon_f = -0.000216 w_f^2 - 0.00604 w_f + 0.0518 \qquad \ldots\ldots\ldots\ldots...(6)$$

Where :

ε_f is the flexural strain (mm/mm)

Analytically equations (obtained from experimental results) befitting with other researchers[Luo and Netravali 1999, Khalid *et al.* 2003]

Conclusions

The tensile and flexural properties of epoxy composites reinforced with rice husk fillers have been studied and discussed here. The following conclusions can be drawn from the present study.

1. The tensile and flexural strengths of the epoxy rice husk filler composites were affected by the amount of filler in the composites.
2. In tensile testing, filler composites demonstrated linear behavior with sharp fracture.
3. In flexural testing, filler composites demonstrated slightly nonlinear behavior prior to sharp fracture.
4. In tensile and flexural tests, the strains decrease with the increase in the filler contents due to the fact that the materials have become harder with the increase in filler contents
5. Analytical relation between maximum stress and filler contents are linear whereas, for modulus of elasticity and strain are quadratic.

References :-

A.A. Khalid, B. Sahari, and Y.A. Khalid, (2003) ,"Environmental Effects on the Progressive Crushing of Cotton and Glass Fiber/Epoxy Composite Cones", Proceedings of the Fourth International Conference on Advances in Materials and Processing Technologies, Tokyo , pp. 680–689 .

A, Maulida, M. Nasir, and H.P.S.A. Khalil, (2000), "Hybrid Composites Based on Natural Fiber", Proceedings of Symposium on Polymeric Materials, Penang, Published by USM Press, Penang , pp. 216–219.

ASTM International ,(2002), Standard Test Method for Tensile Property of Plastics, American Society of Testing Materials (ASTM) , pp. 100–109.

ASTM International ,(2007), Standard Test Method for Flexural Properties of Unreinforced and reinforced Plastics and Electrical Insulating materials , American Society of Testing Materials (ASTM) , pp. 88–95.

C. A. May and G. Y. Tanaka, (1983), "Epoxy Resin Chemistry and Technology". New York: Marcel Dekker.

E.M. Ahmed, B. Sahari, and P. Pedersen, (2003), "Non Linear Behavior of Unidirectional Filament Wound COTFRP, CFRP, and GFRP Composites", Proceedings of World Engineering Congress, Mechanical and Manufacturing Engineering, Kuala Lumpur , pp. 537–543.

E. M. Yorkitis, (1994), “Rubber-Modified Thermoset Resins”, (ed. K. Riew and J. K. Gillham), Advances in Chemistry Series No. 208. Washington, DC: American Chemical Society, p. 137.

H. Shangjin, S. Keyu, B. Jie, Z. Zengkun, L. Liang, D. Zongjie and Z. Baolong, (2004), “Studies on the Properties of Epoxy Resins Modified with Chain-Extended Ureas” , The Arabian Journal for Science and Engineering , 28(2B) , pp. 9641–9647.

J.P. Schneider and A.C. Karmaker, , (1996) ,J. Mater. Sc., 15 , pp. 201.

M. Cazaurang, P. Herrera, I. Gonzalez, and V.M. Aguilar, (2001) , “Physical and Mechanical Properties of Henequen Fibers”, Journal of Applied Polymer Sciences, 53 , pp. 749–756.

M.Y.A. Fuad, S. Rahmad, and M.R.N., Azlan, (2004), “Filler Content Determination of Bio-Based Thermoplastics Composites by Thermogravimetric Analysis” , Journal of Polymer, 51 (8) , pp. 268–275.

P. A. Herrara-Franco, Valadez-Gonzalez, and M. Cervantes, (2002), “Development and Characterization of a HDPE–Sand–Natural Fiber Composite”, Composites Part B: Engineering, 28B (3) pp. 331–343.

R. S. Drake, D. R. Egan, and W. T. Murphy , (1992) ,“Epoxy Resin Chemistry II”, (ed. R. S. Bauer), ACS Symposium Series No. 221. Washington, DC: American Chemical Society , p. 1.

S. Luo and A.N. Netravali, (1999), “Mechanical and Thermal Properties of Environmentally Friendly “Green” Composites Made from Pineapple Leaf Fibers and Poly (hydroxybutyrate-co-valerate) Resin”, Polymer Composites, 20 (3) , pp. 367–378.

Z. Zhikai, Z. Sixun, H. Jinyu C. Xingguo, G. Qipeng, and W. Jun, (2002), "Phase Behavior and Mechanical Properties of Epoxy Resin Containing Phenolphthalein Poly ether ether Ketone", Journal of Polymer, 44 (5) , pp. 1075–1080.

Table (1) physical properties of used epoxy resin

Property	Unit	Value
Viscosity	Pa.s	0.85
Density	g / cm^3	1.2
Water absorption	%	1.0

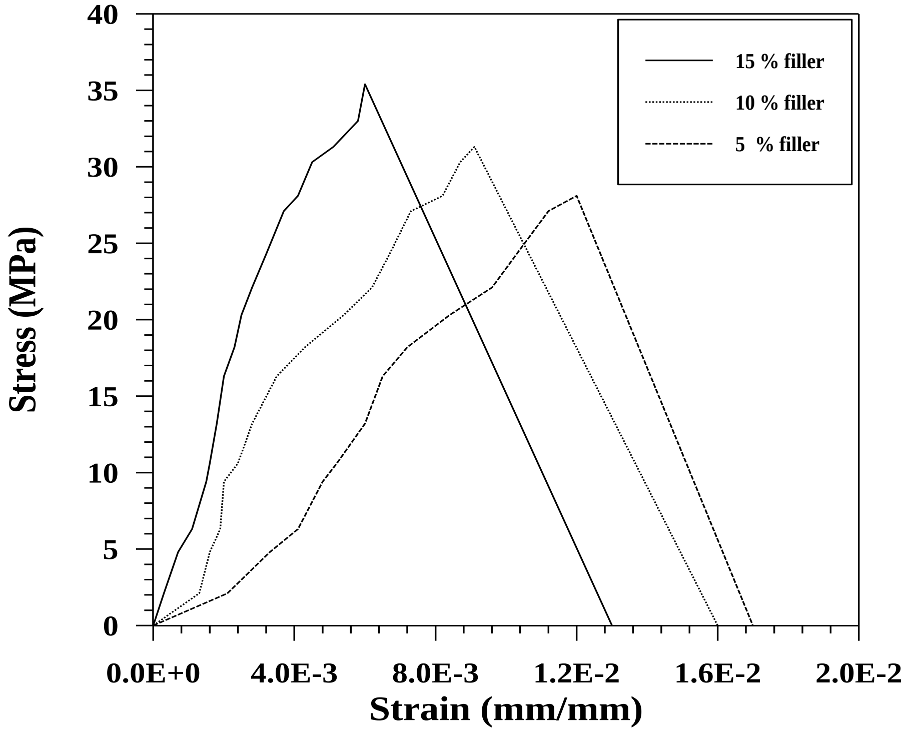

Figure 1. Show the relation between tensile stress and tensile strain for composite material

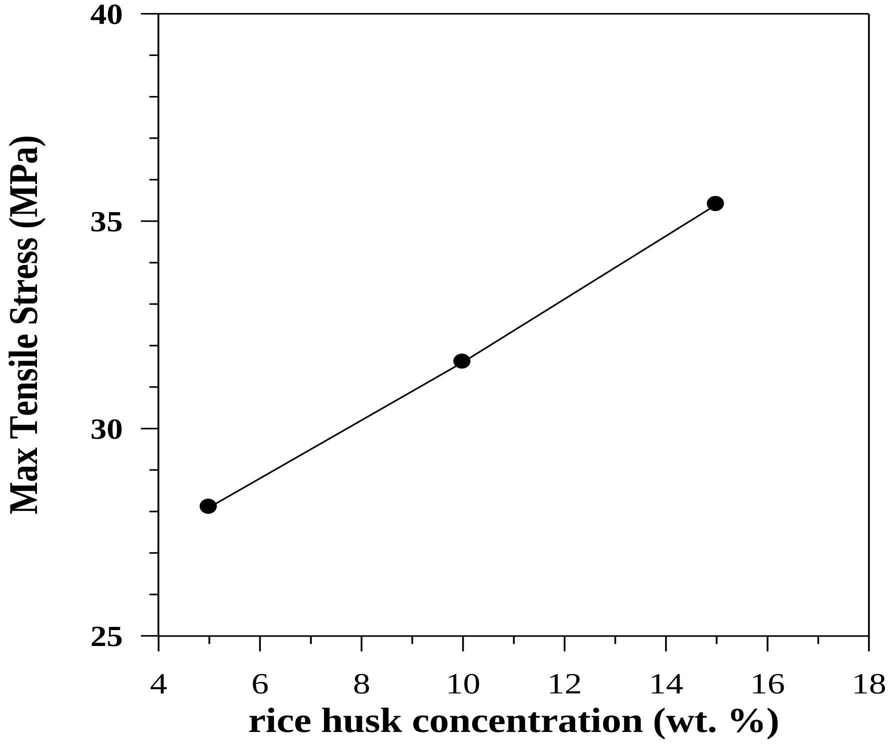

Figure 2. Show relation between max tensile stress versus rice husk concentration

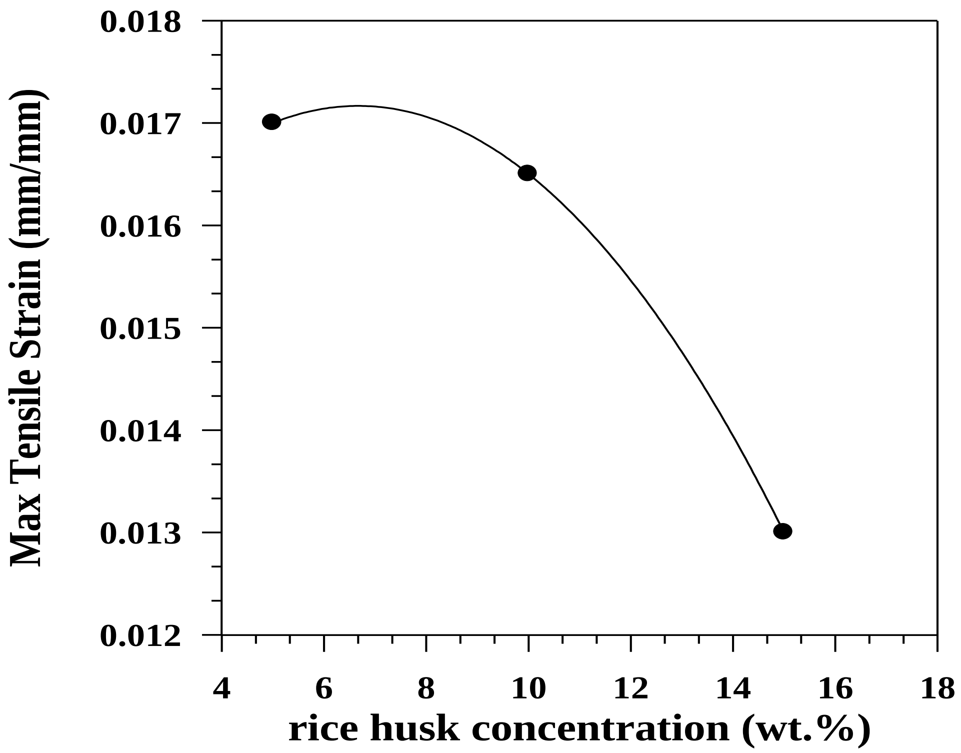

Figure 3. Show relation between max tensile strain versus rice husk concentration

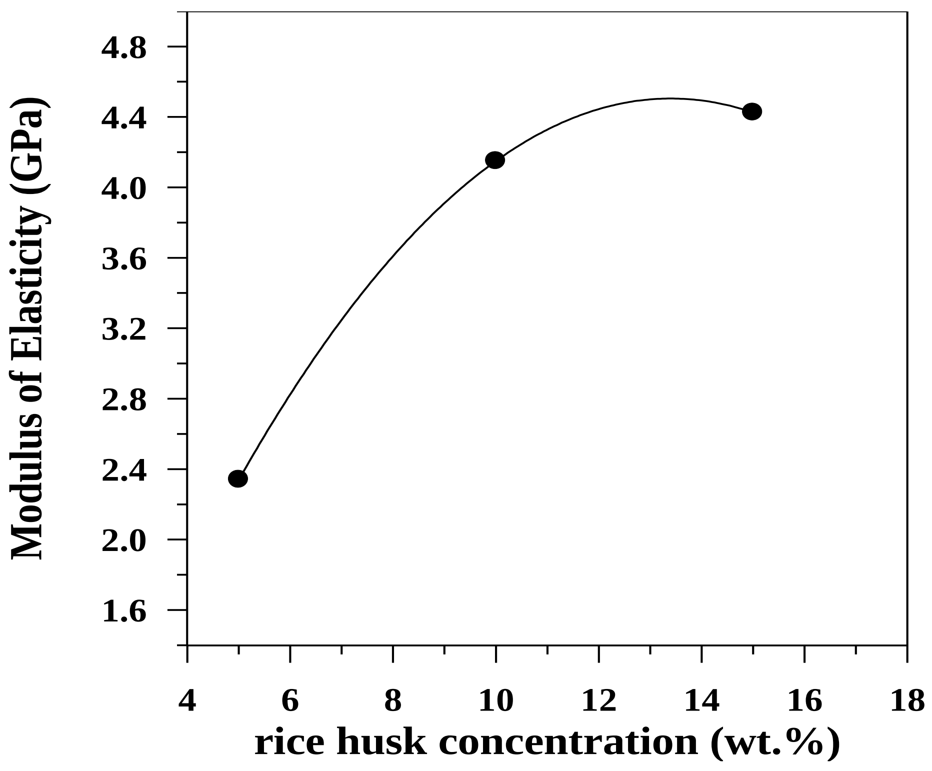

Figure 4. Show relation between tensile modulus of elasticity versus rice husk concentration

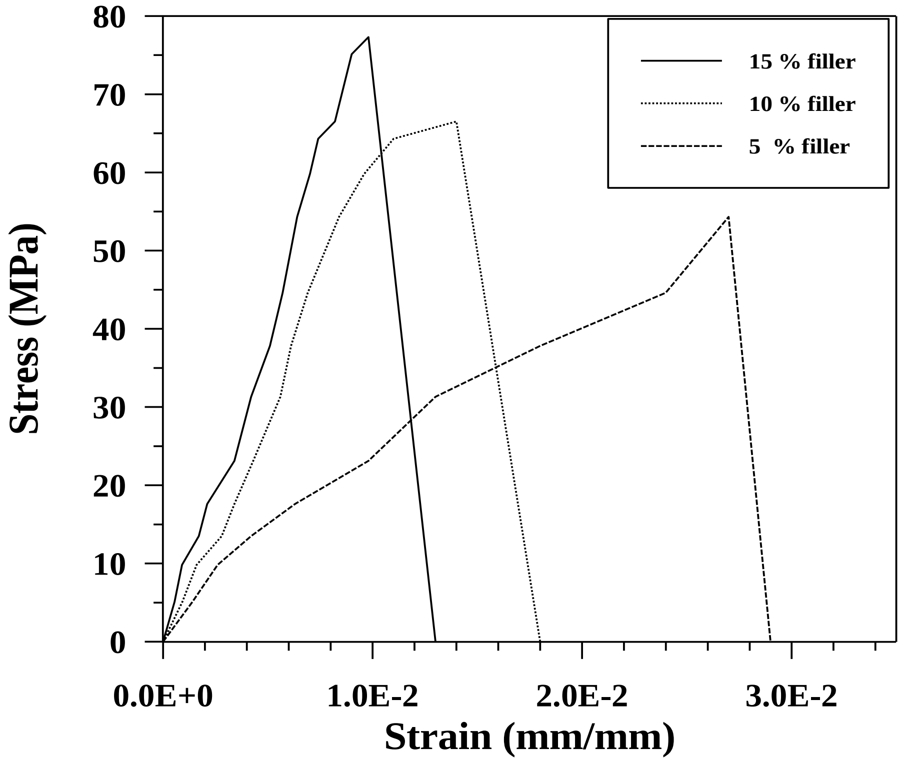

Figure 5. Show the relation between flexural stress and flexural strain for composite material

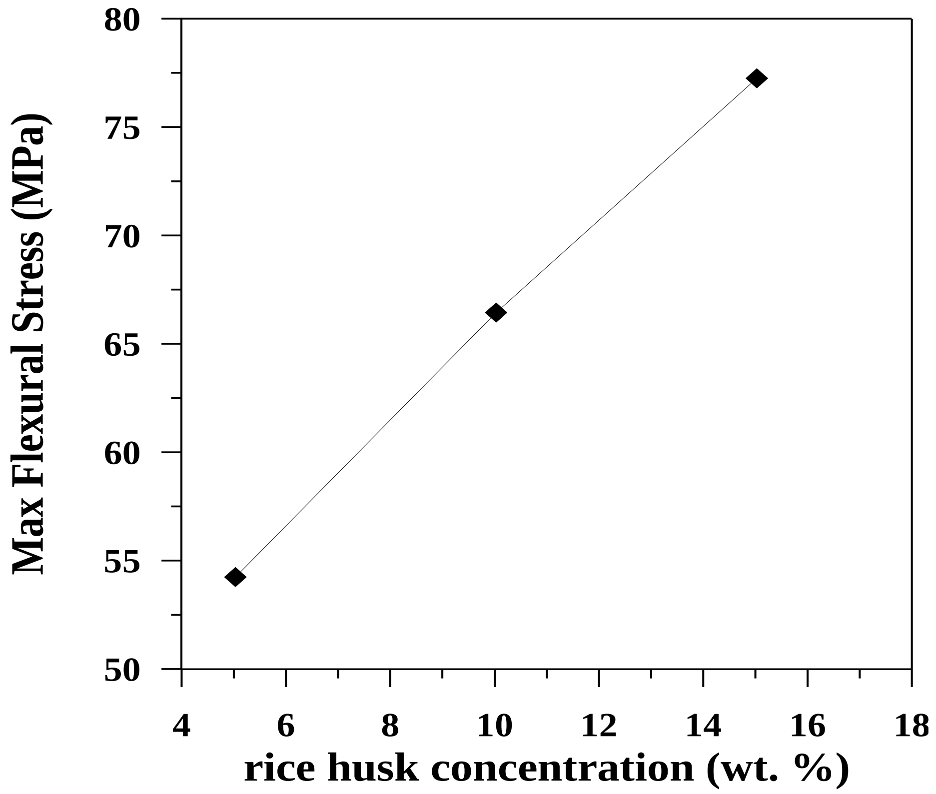

Figure 6. Show relation between max flexural stress versus rice husk concentration

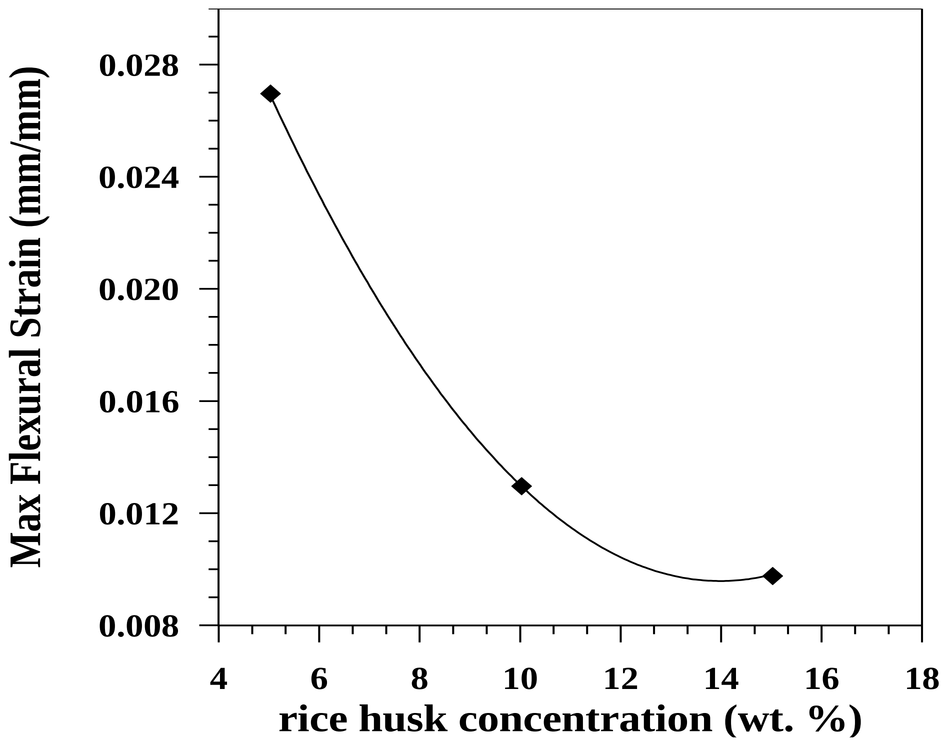

Figure 7. Show relation between max flexural strain versus rice husk concentration

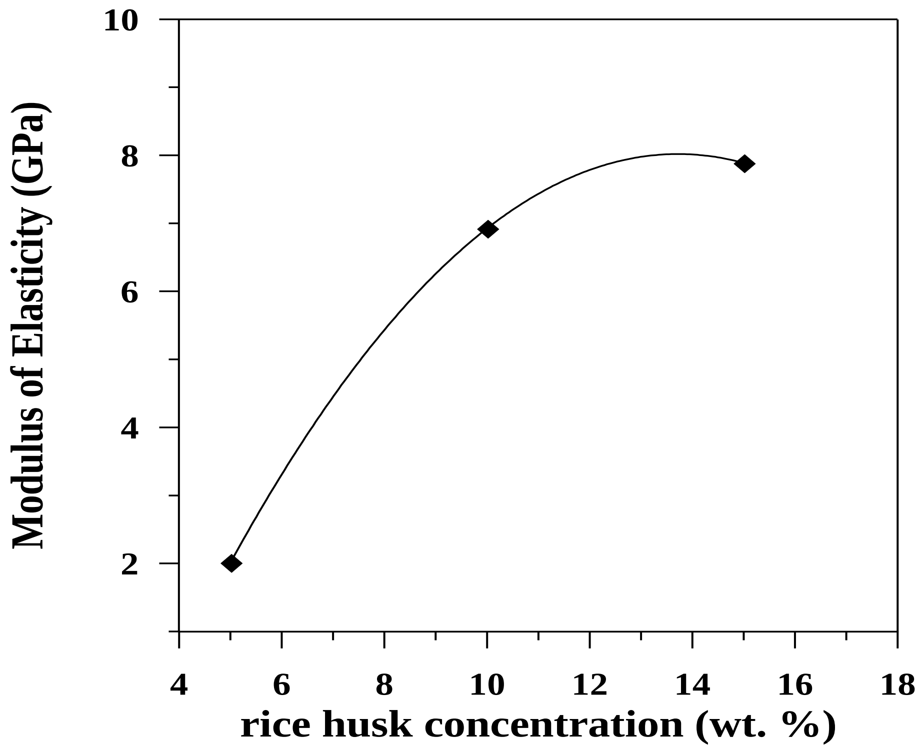

Figure 8. Show relation between flexural modulus of elasticity versus rice husk concentration