The Mechanical and Thermal Properties of Polymer Hybrid Composite Reinforced by Rice Husk Ash at Different Conditions

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ABSTRACT

This research was carried out to study the effect of natural and calcined temperature of rice husk ash with selected temperature (900,1100,1300^{o}C) on the mechanical and thermal properties for the prepared hybrid composite with fixed volume fraction (25% fiber glass, 5% rice husk ash).The mecha
nical properties include (tensile strength, bending strength, impact strength and hardness). The results show that the mechanical and thermal properties increased with increasing the calcined temperature and the best calcining temperature of rice husk ash is 1300^{o}C, grinding rice husk before burning led to a decrease in mechanical properties and thermal conductivity.

Keywords: Hybrid composite.

الخواص الميكانيكية والحرارية لمتراكبات هجينية بوليمرية مقواة برماد قشور الرز بظروف مختلفة

الخلاصة

يهدف البحث دراسة تأثير طبيعة رماد قشور الرز ودرجة حرارة الحرق المختارة **(900,1100 and 1300^{o}C)**على الخواص الميكانيكية و التوصيلية الحرارية للمترابكات الهجينة المحضرة بنسب حجمية ثابتة (5% رماد قشور الرز و 25% ألياف الزجاج)تضمنت الخواص الميكانيكية (مقاومة الشد ,مقاومة الانحناء , الصلادة و مقاومة الصدمة). وقد بينة الدراسة زيادة الخواص الميكانيكية وكذلك التوصيلية الحرارية مع زيادة درجة حرارة الحرق و ان طحن القشور قبل الحرق ادى الى انخفاض الخواص الميكانيكية والتوصيلية الحرارية .

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INTRODUCTION

Currently material scientists around the world are engaged in the synthesis of new green polymeric material and processes that improve the environmental quality of a number of products [1-5]. Over the past two decades organic filler have becoming strong competitors to this traditional inorganic filler due to their low densities, very low cost, non-abrasiveness, possibility of high filling levels, recyclability, biodegradability and renewable nature [6].

Rice husk (Rh) is one of the major agricultural residues produced as a by-product during rice milling process. Usually it has been problem for rice farmers due to its resistance to decomposition in the ground, difficult digestion and low nutritional value of animals [6]. According to Marti-ferrer the lignin and hemicellulose contents of rice husk are lower than wood whereas the cellulose content is similar, for this reason rice husk flour (RHF) can be processed at higher temperature than wood, there for the use of rice husk in the manufacture of polymer composites is attracting much attention [7].

Group of researchers have published many studies dealing with polymer-rice husk composition [1, 8-10]. They observed that tensile and impact strength decreased with increasing filler content while the elastic modulus increased [1, 9]. Despite the low properties of the composites the researchers concluded that rice husk flour could be utilized as a biodegradable filler to minimize environmental pollution [1]. On the other hand another study has been focused mainly on the improvements in the thermal stability, dynamic mechanical properties, tensile properties and crystallinity with addition of maleic anhydride-grafted PP (MAPP) [10-11]. The main components of rice husk are cellulose (25%- 35%), hemicelluloses (18%- 25%), lignin (20%- 31%), ash 17% (silica 94%), soluble (2%- 5%) and moisture 7.5% [4, 6, 11-13]. The rice husk ash (RHA) which is produced by burned of rice husks has a high content of silica (95% 1300^{o}C- 99% silica by mass) with minor amounts of metallic elements. This rice husk ash can be an economically viable raw material for the production of silicates and silica, and may be used as a filler of different polymers [13-14].

In the present study it was used a thermosetting polymer (Epoxy resin) as matrix and rice husk ash with fiber-glass as filler to prepare hybrid composites. The objective of the work was to explore the effect of burning temperature and pre-ground of raw rice husks on the mechanical properties and thermal conductivity behavior.

Procedure:-

Material which used in this research prepared from epoxy resins as a matrix material and fiber glass and rice husk ash as areinforced materials. Epoxy resin which had been used is (Quick mast 105) produced from don construction products LTD. (D. C. P.) and it is characteristic as transparent liquid at room temperature converted to a solid state by adding suitable hardener. Hardener regarded as transparent liquid which is added to the epoxy resin in the ratio (1:3) and then the produced mixture must be blending continuously for (10-15) minutes in order to get good homogenous mixture. Some of mechanical and physical properties of the used epoxy resin referred in the table (1).

Table (1)some of mechanical and physical properties of epoxy resin.

Compressive strength at 20°*C*for 7 days	Flexural strength at 35°*C*	Tensile strength	Specific gravity	Viscosity at 35°*C*
72 N/mm²	60 N/mm²	25 N/mm²	1.04	1.0 poise

Fiber glass which used as arienforced material in this research is from E-glass type in the form of chopped strand mat produced from Mowding L.T.D. /UK. Company with diameter range (4-6) μ*m*. Table (2) refers to the chemical composition of the used fiber glass, while table (3) refers to some of mechanical and physical properties of used fiber.

Table (2)The chemical composition of fiber glass

Material	SiO_2	CaO	Al_2O_3	MgO	B_2O_3	TiO_2	Na_2O	Fe_2O_3	F
%	53	22	12.3	3	6	0.5	1.6	0.6	1

Table (3) Mechanical and physical properties.

Water Absorption %	Thermal conductivityw/m.k	Modulus of elasticity GPa	Elongation at Break %	Tensile strength MPa	Density Kg/m³
< 0.1	1	73	2.9	1600	2600

The second reinforced material is rice husk ash which is obtain from -Al-Najaf rice farm, there is series of operations had been carried on the rice husks, at first the rice husk used were taken from rice mills. As The first step the rice husk were washed thoroughly with tap water to remove soils and dirt, and dried in air at the room temperature. The dried husks were washed with distilled water and dried again in an oven at 55°*C*. The dried husks were treated chemically with 20% HCI acid. A proper amount of 1 molar of HCI had been used. The rice husk mixed with 1 molar of HCI in a glass beaker for three hours, and then the solution was filtered and the rice husks were washed with distilled water several times in order to removing the acid. After that these rice husks were dried in oven at 100°*C* for six hours. Then some of the cleaned rice husks were ground in the rotary cutting mill type (branbender Duisburg 880804) to get fine powder and then some of the cleaned rice husk is grounded, and finally the grounded and non-grounded rice husk fired are burned at different temperatures (900- 1100-1300) °*C* in electrical furnace for 2 hours. The chemical composition of rice husks which was burned at 1100°*C* was shown in table (4).

Table (4)the chemical composition of rice husks which was calcined at 1100°C.

Material	Sio2	CaO	Al2O3	K2O	CaO	Na2O	MnO	Fe2O	L.O.T
%	93.4	0.22	0.76	1.17	0.88	0.87	0.79	1.02	0.89

It can be seen from the chemical composition that the ash produced from the rice husks burning contains a great amount of silica and small amount of oxides considered as impurities.

Through this research some of burned rice husk (rice husk ash) had been examined by X-ray fluorescence test which indicate the presence of the following elements in the rice husk ash (R.H.A). (K, Si, Ca, Mn, Fe, Ca and Zn) which referred in the figures. (1), (2) and (3). Differences in composition may occur due to geographical factors, type of ground, year of harvest, and sample preparation and analysis methods [15].

In this research it was studied the effect of the elements which present in the rice husk ash which used as a reinforced material with compensation with the fiber glass on some mechanical and thermal conductivity properties of prepared hybrid composite materials with a total reinforcing material volume fraction of (30%) which contain (25% fiber glass and 5% non-ground or pre-ground rice husk ahs) as indicated in the table (5).

Table (5) the composition of the prepared hybrid composite.

	Vol.% of Epoxy	Vol. % of F.G.	Vol.% of R.H.A.	From of R.H.	Burning Temperature
1	70	25	5	Pre-ground	900°C
2	70	25	5	Pre-ground	1100°C
3	70	25	5	Pre-ground	1300°C
4	70	25	5	Unground	900°C
5	70	25	5	Unground	1100°C
6	70	25	5	Unground	1300°C

Mechanical test:-

Sample were prepared for the tensile test in accordance with ASTM D 638 procedure, instron 1195 tensile test machine, the test was conducted at the constant strain rate of the order of [10mm/min] at room temperature. Tensile stress was applied till the failure of the sample and stress strain curve was obtained. Each sample was tested for three times and average results have been reported. Flexural modulus measured from three point test, this test is preformed according to ASTM D 790-78 at room temperature.

Iozdimpact test is preformed according to ASTM ISO 179 at room temperature. The hardness test carried out on a Durometer on shore-D scale (ASTM D/N-53505) standard is used for hard polymer material with specimen dimension of 40mm diameter and 5mm thick.

Thermal conductivity test

This test had been accomplished by using Lee-Disc instrument, in which the value of thermal conductivity determined by using relation (1) and (2), [16].

$$K\left[\frac{TB-TA}{Ts}\right]=h\left[TA+\frac{2}{r}\left(dA+\frac{1}{2}ds\right)TA+\frac{1}{r}ds\,TB\right]\quad \dots\dots\dots\dots(1)$$

$$H=i.v.=\pi r^2 h(TA+TC)+2\pi rh\left[dA\,TA+ds\frac{TA+TB}{2}+dB\,TB+dC\,TC\right]\dots(2)$$

Where:

h: - Heat loss per second and area of (m^2) and at temperature different between disc and environment, H: - Supplied energy, r: - Disc radius, d: - Disc thickness and TA, TB, and TC: - are the temperature of the discs A, B and C above ambient.

Results and Discussion

The investigation of mechanical properties of composites is one of the most important techniques in studying the behavior of composite material. It has been proved to be the most effective method to study the behavior of the material under various condition of tension, compression stress and phase composition of filler composites and its role in determining the mechanical properties.

Results of tensile test

The tensile strengths and modulus of elasticity of the composites made of (rice husk ash 5% and fiber glass 25%) fillers and epoxy resin matrix at different rice husk burning temperature, for different nature ash (pre-ground and unground) are shown in Figures (4) and (5) respectively, tensile strength and Young's modulus values slightly increased with increasing rice husk burning temperature from 900°*C* to 1300°*C*. This behavior is belonging to the presence of silica in the crystalline form. The crystalline, amorphous silica or both presences is due directly to the temperature of burning or the method of ash attainment. When the burning temperature of rice husk is high, the silica contained in the ash is predominant crystalline [15, 17]. Shinohara and kohyama had quantified the crystalline silica in leached ashes sample gotten by rice husks burning in some temperature and time, and had observed that in temperature around of 1000°*C* it gets ash with predominant crystalline silica, getting 83% of crystalline silica at temperature of 1350°*C*, in the range of temperature of 450°*C* -700°*C*, the silica was presented under the amorphous phase (less than 5% of crystallinity) [17]. And also from these figures it was observed that the unground rice husk gives higher tensile strength and elasticity modulus values compare to the pre-ground rice husk at the same burning temperature, because in the case of non-grounded rice husk these reinforcing material through burning process will change in to small fibers and the fibrous form will increases the effect of reinforcing as compared to the form of the small particles which produced from burning pre-grounded rice husk ash, the worsening interfacial bonding between filler and matrix polymer decreasing the tensile strength. For irregularity shape filler which produced from burning pre-grounded rice husk ash, the strength of the composites decreases due to the inability of the filler to support stresses transferred from the polymer matrix, while poor interfacial bonding causes partially separated micro-spaces between filler and matrix polymer, which obstructs stress propagation when tensile stress is loaded and induce increased brittleness [1].

While figure (6) refer to the effect of the reinforcing material on the ductility of prepared samples, from this figure it can be seen that the ductility will increase gradually with increasing burning temperature of used rice husk ash due to the purity and crystallinity of the produced (silica) which will increases with increasing the burning temperature. As well as the value of ductility of the material which reinforced by ungrounded rice husk ash is higher than that of the material which reinforced by groundrice husk ash and at the same burning temperature.

Result of Bending Test:-

Figure (7) refers to the effect of the burning temperature on the value of flexural modulus (E_{bend}) of prepared samples, this figure shows the value of (E_{bend}) will increase for each sample with increasing the burning temperature due to the formation of amorphous silica in the burning temperature range (450- 750)°*C* [18] and when burning temperature increased above (750°*C*) the degree of crystallinity of the produced (SiO2) will increase and becomes clear at temperature about (1000°*C*). Above (1000°*C*) the produced quartzes becomes more purity and at (1300°*C*) this produced ceramic material will be in high degree of purity and crystallinity [17].This phase transformation process of produced (SiO2) and which associated with purity and crystallinity degree increasing will lead to increase the (E_{bend}) of the prepared samples with increasing burning temperature, from the same figure, it can be shown that the values of (E_{bend}) of the samples which reinforced by non-ground rice husk ash is higher than that of the samples which reinforced by pre-ground rice husk ash, this is due to the formation of fibrous particles from the ashes of the rice husks non milled, and it will appear later in the optical micrograph.

Result of Impact Test:-

Figures (8) and (9) refer to the effect of the burning temperature of rice husk on the values of impact strength and fracture toughness respectively of prepared hybrid composite samples with (25% fiber glass + 5% rice husk ash), from these figures it is observed that these values will increase with increasing burning temperature up to (900°*C*) due to phase transformation process which is carried on the produced (SiO2), the purity and crystallinity degree will increase with increasing burning temperature of rice husk ash [15],and from the same figures it can be seen that the impact strength and fracture toughness of the non-ground rice husk ash rienforced hybrid composite material is higher than that of the pre-groundrice husk ash, that related to the non –ground rice husk, which will be change in to small fibrils during burning process which has good ability for supporting the applied load on the prepared composite material while the ground rice husk will change to small particles during burning process. So it is important to say that the energy which is required to pull out the fiber from matrix is much higher that the energy which is required to pull out the particles from matrix and hence this will rising from the stress which required to failing the prepared composite material.

Results of Hardness Test:-

The value of hardness of the prepared samples had been determined by using (shore-D) instrument; figure (10) indicates that the values of the hardness will increase with increasing burning temperature of rice husk ash due to the nature of rice husk which is converted from amorphous structure (silica) to crystalline structure (quartz) of higher hardness. The same figure show that the value of the hardness of the un-ground rice husk ash rienforced composite materials higher than the hardness of the pre-ground rice husk ash rienforced composite materials and this is because that in the case of using non-ground rice husk ash rienforcing material the bonding force between fibers and the matrix is higher than the bonding force between the particles and the matrix when the material rienforced by ground rice husk ash.

Results of thermal conductivity test:-

The results of the thermal conductivity test which is carried on the prepared samples shown in Figure (11), from this figure it was observed that the increasing in the burning temperature of the used rice husk will lead to increased thermal conductivity for prepared samples due to phase transformation of rice husk which will lead to increasing the degree of purity and crystallinity of produced (SiO_2) [12] and hence the value of thermal conductivity will increase.

Figure (11) is also refer to the effect of the shape of rice husk ash which is used as arienforcing phase on the thermal conductivity of the produced composite materials, from this figure it can be seen that the thermal conductivity of non-ground (fiber form) rice husk ash reinforced material will be higher than the value of thermal conductivity of the ground (particle form) rice husk ash reinforced material and this is because the fiber form of non-ground rice husk ash will has high bonding force at the interface area and hence the percentage of the porosity will decrease, so the value of the thermal conductivity will increase.

Microscopic examination

Figure (12) shows the microstructure of the samples reinforced bynon-ground rice husks and pre- burned at different temperature (900, 1100, 1300 ^oC) notes from the figures (a),(b) and (c) that the microscopic structure of the rice husks ash appear as fibrous form after the burn at 900^oC as shown in figure (a), and when increase burn temperature to 1100^oC and 1300^oC that lead to greater the ratio of the fibrous structure for the rice husks ash (figures a,b) and this led to improve the mechanical properties of the prepared composite material.

Conclusion:

Depending upon the results of the tests samples which carried on this research it was concluding that:-

1. Increasing the burning temperature of the used rice husk ash lead to increase the mechanical properties (tensile strength, young's and, flexural modulus, impact strength, fracture toughness and hardness) and thermal conductivity of the prepared hybrid composite materials.

2. Reinforcing by unground rice husk ash gave results much higher and better than that when the materials reinforced by pre-ground rice husk ash at the same volume fraction for all mechanical properties and thermal conductivity property.

3. The highest values of fracture strength, impact strength and fracture toughness reach to 35.77MPa, 70.3 KJ/m2 and 60.57MP$\sqrt{m}$ respectively.

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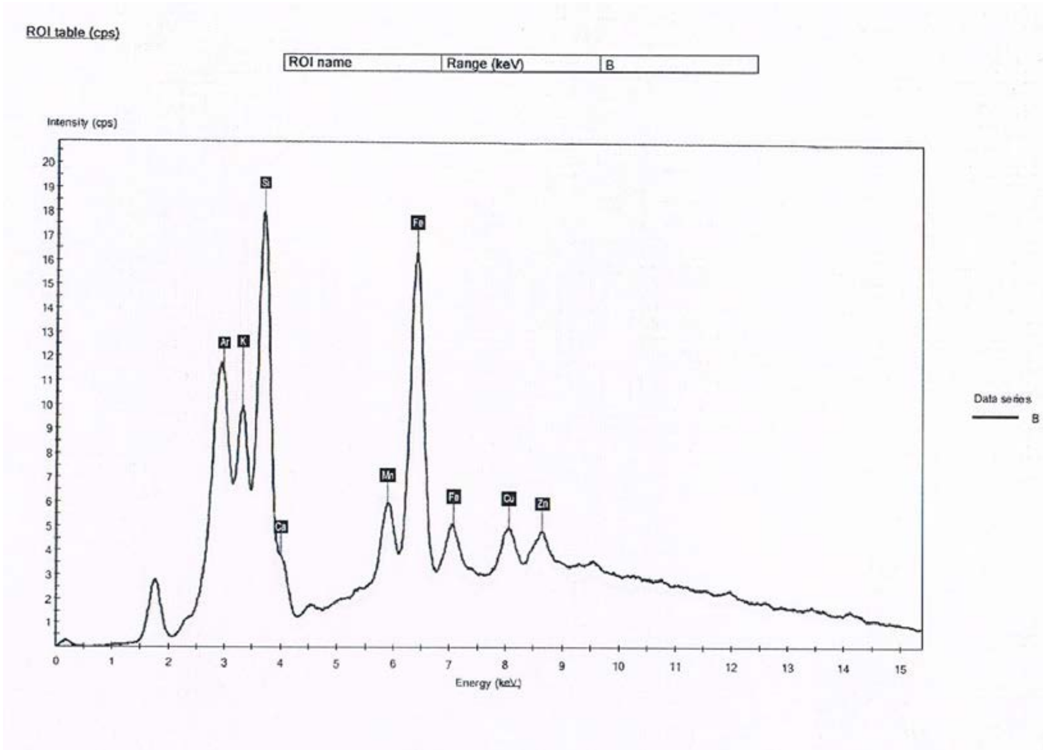


Figure (1):- (XRF) test for rice husk burned at 900°*C.*

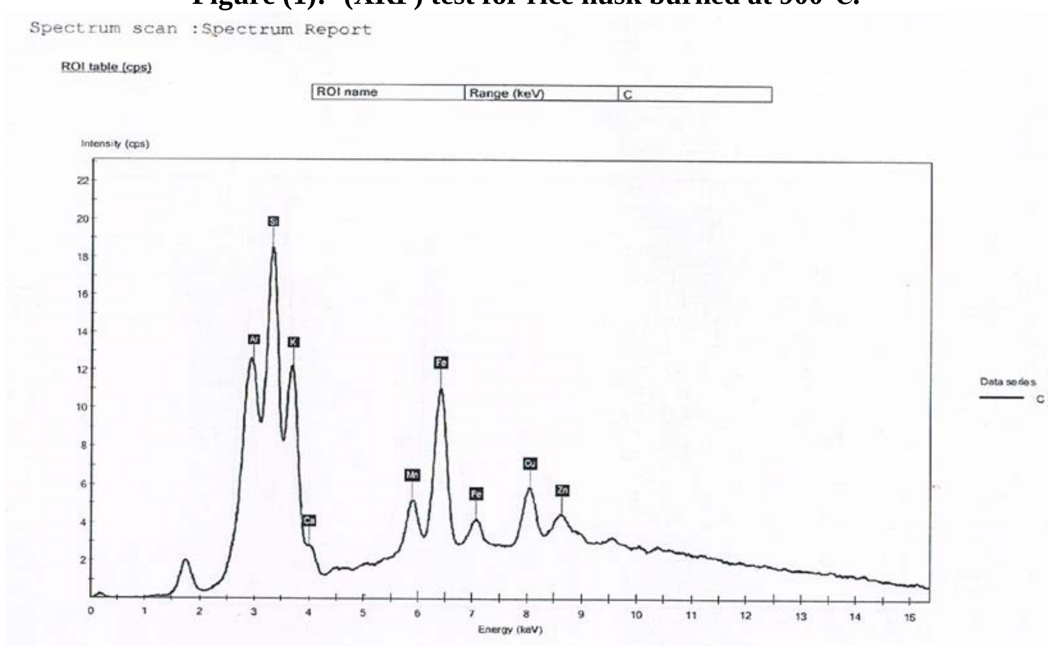


Figure (2): (XRF) test for rice husk burned at 1100°C.

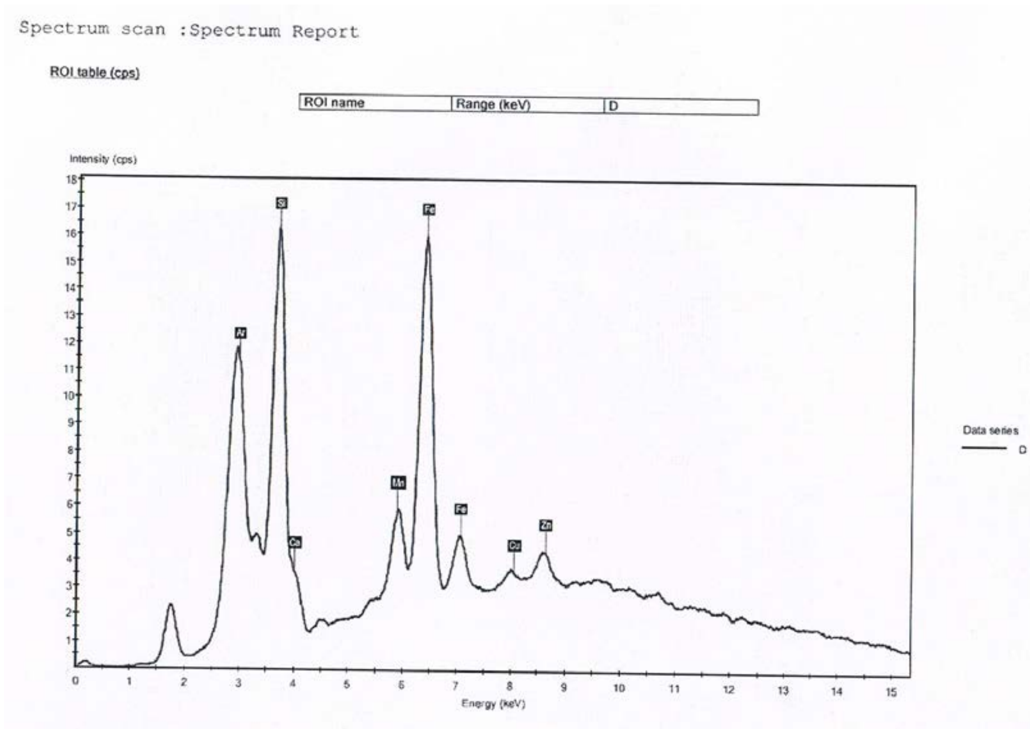


Figure (3): (XRF) test for rice husk burned at 1300°C.

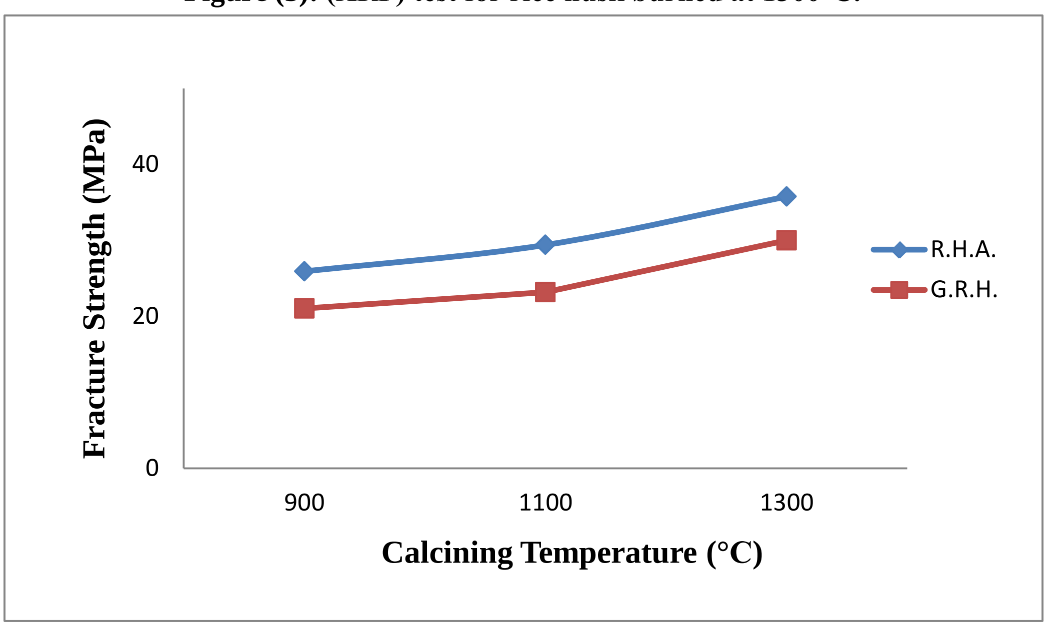


Figure (4): The relation between stress at fracture point and calcining temperatures.

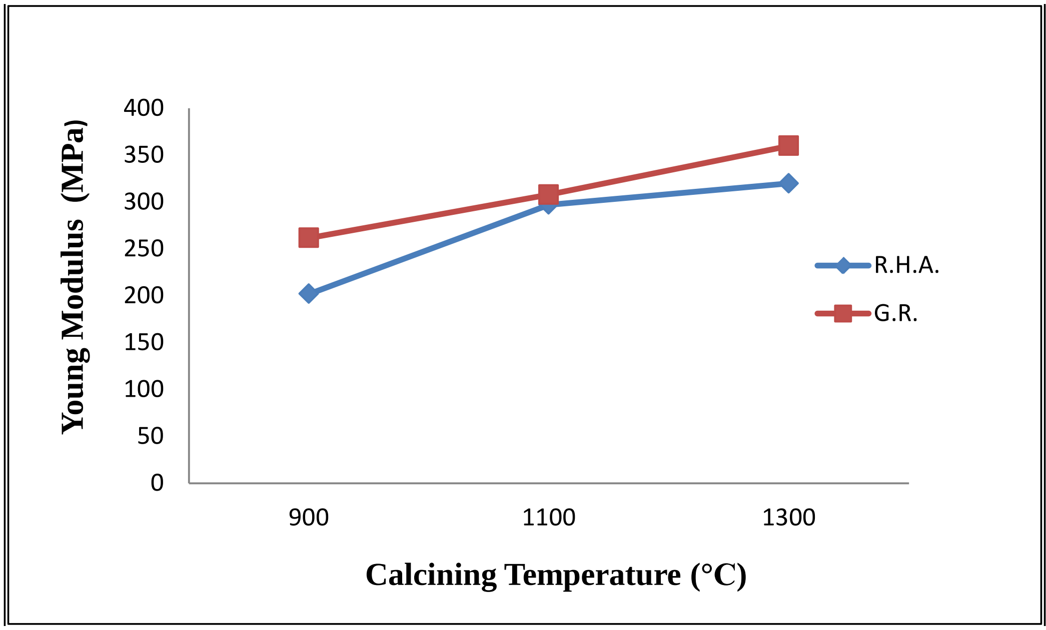


Figure (5):The relation between young modulus of elasticity and calcining temperatures.

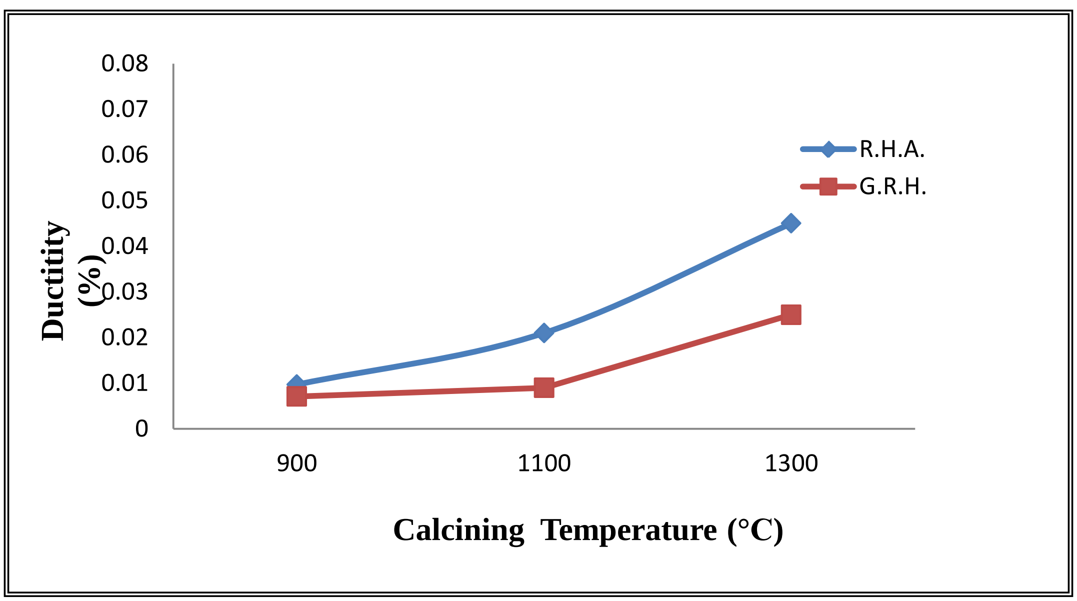


Figure (6):The relation between ductility and calcining temperatures.

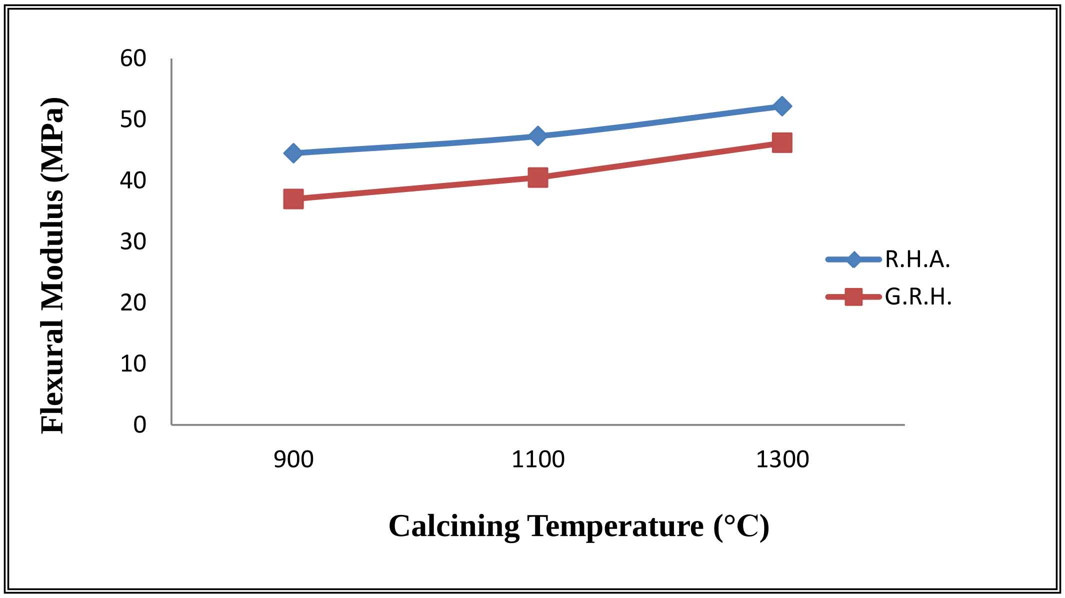


Figure (7):The relation between flexural modulus(E_{bend}) and Calcining temperatures.

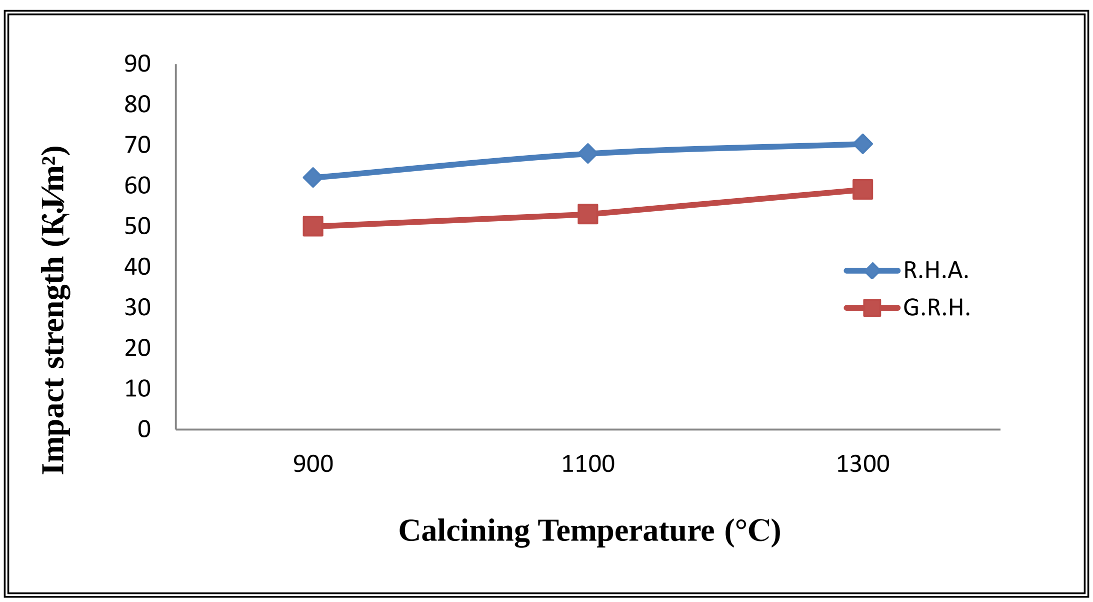


Figure (8):-The relation between impact strength and Calcining temperatures.

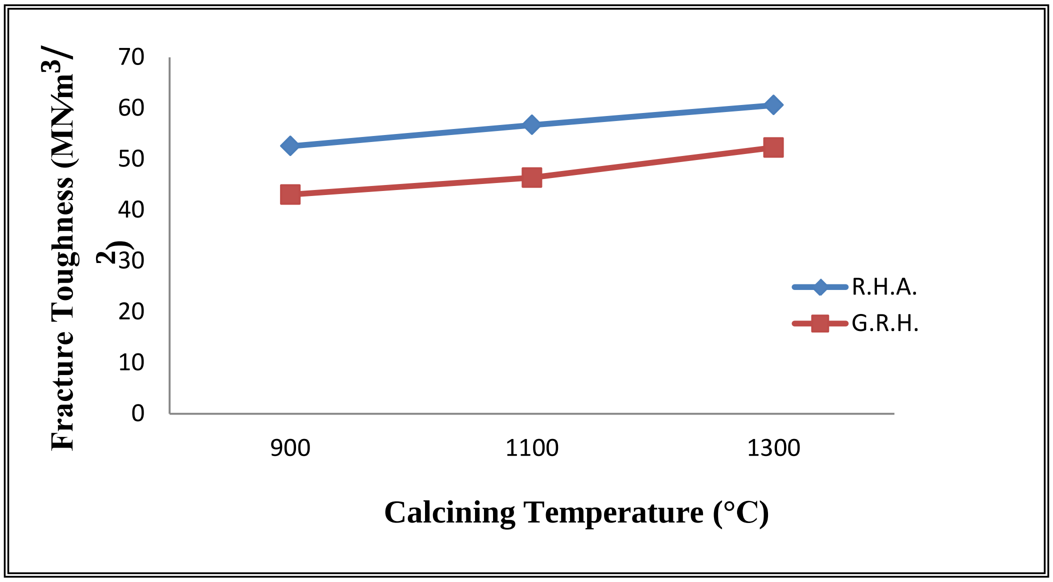


Figure (9):-The relation between impact fracture toughness and Calcining temperatures.

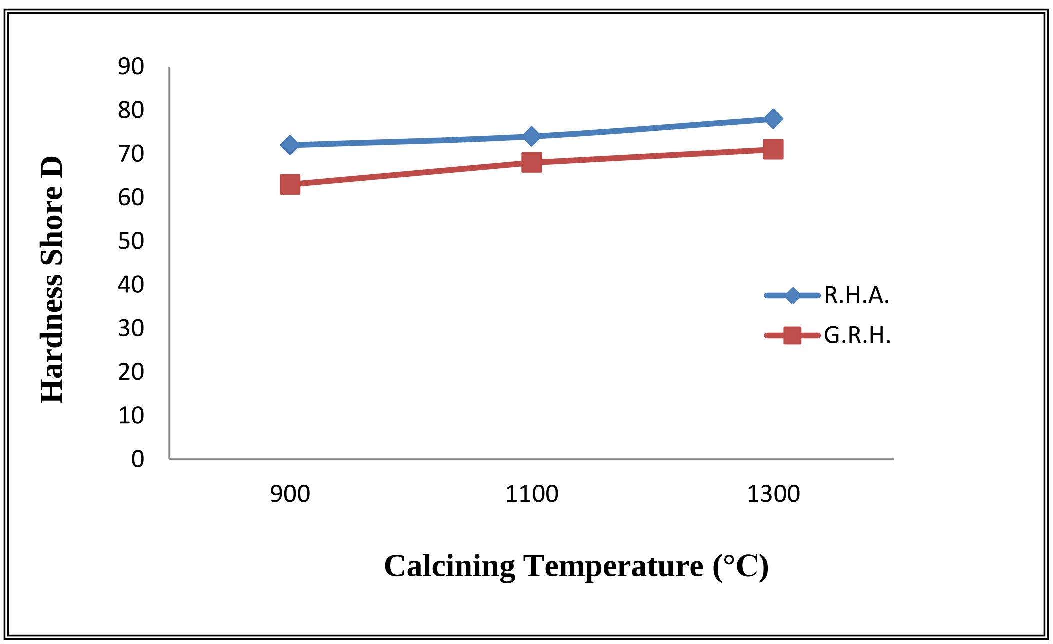
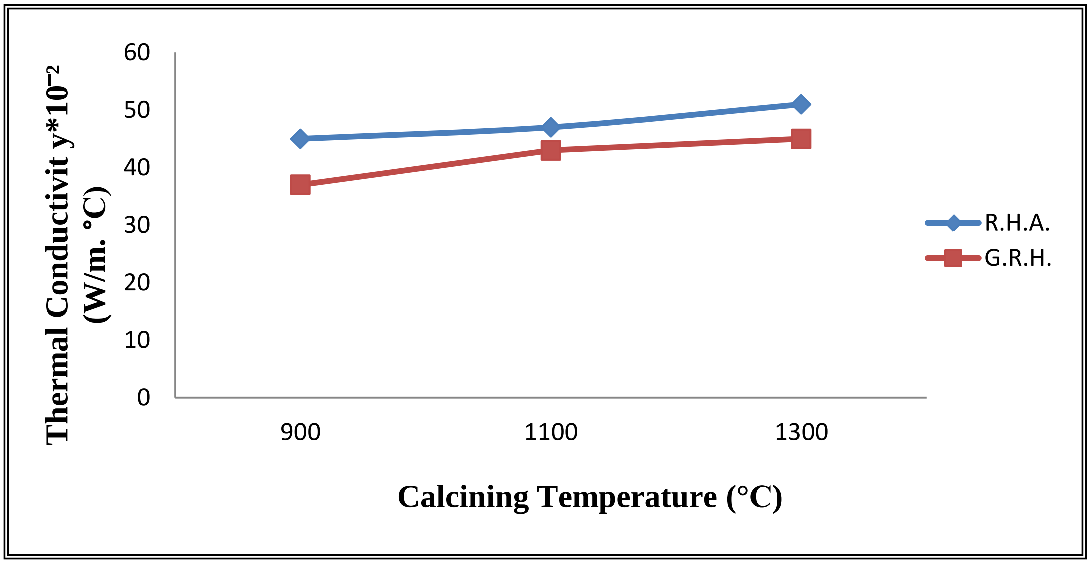


Figure (10):-The relation between hardness and Calcining temperatures.



Figure(11):-The relation between thermal conductivity and Calcining temperatures.

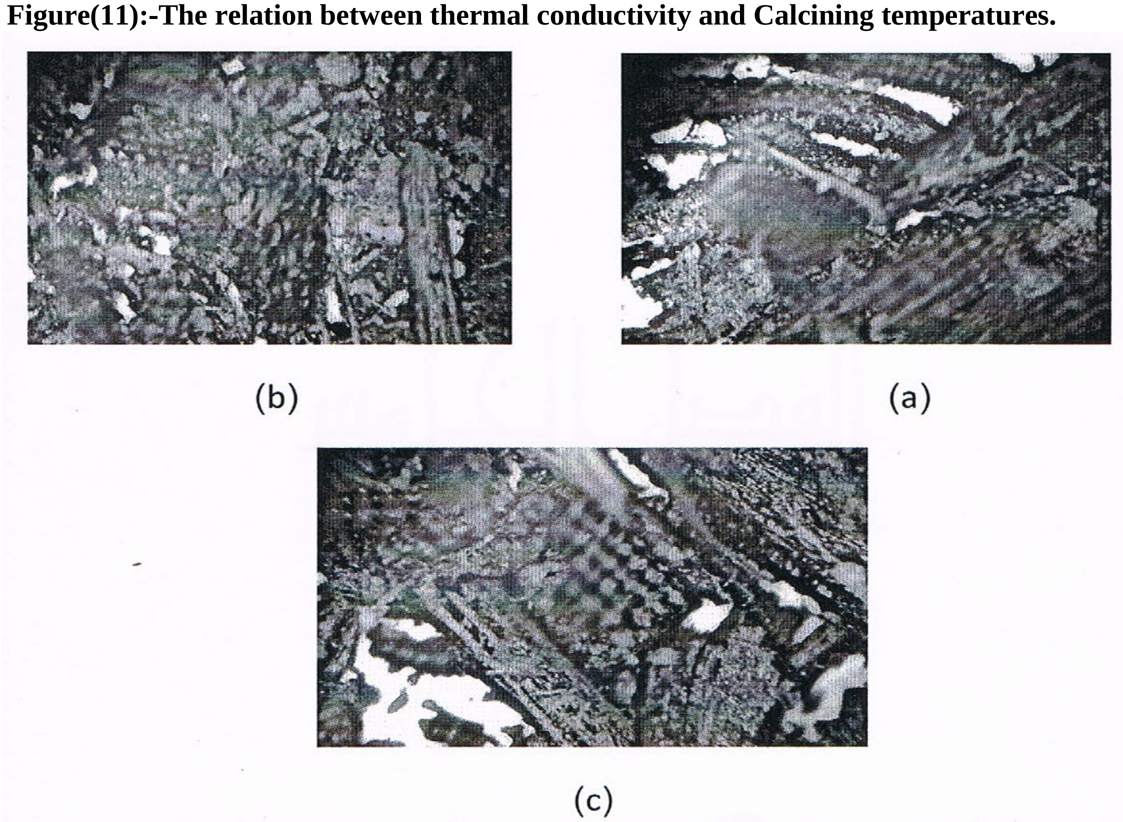


Figure (12): the optical micrograph of rice husk samples calcined at (a) 900°*C* (b) 1100°*C* (c)1300°*C*