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Effect of Rice Husk Reinforcement on Low Density Polyethylene

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Abstract. Low density polyethylene (LDPE) is one of the most common commodity thermoplastics and covers a broad range of applications whereas rice husk is the coating on a seed or grain of rice. Different studies have been done to show the effect of rice husk on high density polyethylene and linear low density polyethylene the limited data is available that shows the effect of rice husk on the properties of LDPE. In this research, rice husk is reinforced in LDPE by using two roll mills as mixing equipment for uniform mixing, the roller is heated at a temperature of 120°C and the particle size of the rice husk is 60 μm. The uniformly mixed material is converted into the shape of the sheet by using a hot press machine. The temperature of the mold is 160°C and the pressure is about 200 psi. The sample is then cooled at room temperature. The sample was characterized by tensile testing, tear testing, hardness testing, thermal gravimetric analysis and Fourier Transform Infrared Spectroscopy. According to the results, the thermal stability of reinforced material is increased as compared to pure LDPE. The hardness, tensile strength and modulus were also increased with a decrease in elongation and tear resistance. Fourier Transform Infrared Spectroscopy results indicate no change in the chemical properties of the matrix, the composite formed was just a physical mixture of rice husk. Overall the reinforcement of rice husk in LDPE increased the thermal stability and mechanical properties of the sample. The cost of rice husk is also lower than LDPE. So, the addition of rice husk will also decrease the overall cost of the product.

Keywords: Fourier transform infrared spectroscopy, hardness testing, low density polyethylene, rice husk, tensile testing, tear testing, thermal gravimetric analysis.

Introduction

Polyethylene is prepared by the polymerization of ethylene in the thermoplastic resin form. It is a slightly crystalline and slightly amorphous polymer those properties generally change as the crystallinity level is constrained by the number of branches on fundamental polymer chains. As the branching is diminished, the level of crystallinity rises. The polymerization controls branching and crystallinity whereas a high temperature process is utilized to deliver polymers of medium crystallinity. (Chen *et al*., 2016; Majeed *et al*., 2014). The liquefy temperature of exceptionally branched polyethylene material is lower than closed packed. In crystalline material, some intermolecular attractions occur in the open structure, the vitality that permits the atom to move separately is lower. Three general kinds of polyethylene are LDPE (low density polyethylene), HDPE (high density polyethylene) and LLDPE (linear low density polyethylene) (Rajendran *et al*., 2018).

The kind of polyethylene formed at high pressure and high temperature polymerization condition is called low density polyethylene. The low density is due to the polymerization condition offers to ascend to the development of numerous branches which are frequently tranquil long to keep the particles from packing close to one another to make a crystalline structure, the structure is predominantly amorphous. LDPE is quite flexible with high impact strength and relatively lower heat resistance (Zhang *et al*., 2019; Zhang *et al*., 2017).

The polymerization condition that results in restricted branching (low pressure and temperature), an increasingly linear polyethylene with just a couple of short branches are made. This kind of polyethylene is called high density polyethylene. The polymer chains in HDPE can simply pack firmly and create crystalline structures as a result the increasing in the density. Generally, HDPE is stiffer which is more grounded and more abrasion resistant than LDPE. To get long polymer chains under the HDPE conditions, a procedure that requires a lot of lower temperatures and lower pressures than the procedure used to make LDPE, a catalyst is required. The principal catalyst was created by Karl Ziegler and was then applied to the polymerization of

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monomers by G. Natta which is called the Ziegler Natta catalyst (Mohamed *et al*., 2019; Yeh *et al*., 2015) . LLDPE is portrayed by its direct structure and the note worthy non attendance of long chain spreading like LDPE. LLDPE has a higher thickness as is progressively crystalline as contrasted to LDPE. The properties of LLDPE can be changed by altering the recipe constituents and the general generation process for LLDPE is usually less essential than LDPE (Zhang *et al*., 2017.

The main reason for choosing this raw material is because LDPE is the most widely used commodity plastic, recyclable high chemical resistance, transparency and low specific gravity. The properties of LDPE are semi inflexible, translucent, extremely intense, great chemical resistance, low water assimilation and effectively handled by most techniques and cables. LDPE's utilizationis to press bottles, toys, transporter packs, high recurrence protection, compound tank linings, hard core sacks, general bundling, gas and water pipes and insulation on electrical wires and cables. Numerous things made with LDPE are gathered for recycling in networks the country over. Inflexible LDPE items (bottles, compartments, covers, tops) are normally gathered in curbside recycling programs (Norhasnan *et al*., 2021).

The rice husk is the covering of a seed or grain of rice. Rice husk has many names, the most widely recognized being husk, chaff and hull. It is formed from hard materials, including silica and lignin to secure the seed in the growing season. Each kilogram of handled white rice commonly achieves 0.28 Kg of rice husk as a rice creation reaction during processing, as appeared in Fig. 1. On normal, 28% of the rice paddy is a husk, giving a yearly all out worldwide creation of 212.5 million tons. It is light weight, having a ground bulk density of 340 - 400 Kg/m^3. It is some what bigger than a grain of rice hence lengths upto 7 mm are conceivable. Common measurements are 4mm by 6mm.This research aims to analyze the change in thermal, mechanical and physical properties of LDPE incorporated with rice husk. Rice husk is used with LDPE to improve the thermal properties which reduce the weight, improve mechanical properties, and reduce LDPE costs (Chun *et al*., 2016; Arjmandi *et al*., 2015).

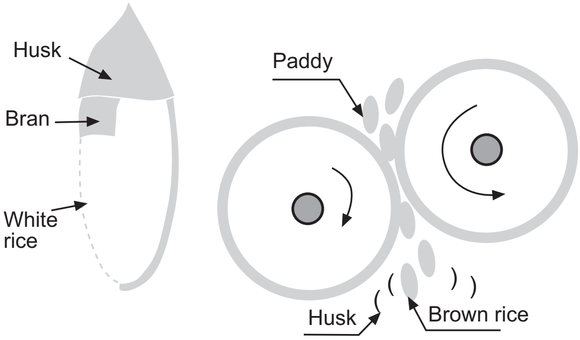

Fig. 1. Rice husk crushing.

Rice, maize and wheat are the three most developed yields on the earth, with rice being the most generally eaten day by day staple for more than 2/3 of the total populace. In 2018 roughly 5.4 million tons of raw husk were accessible from Vietnam alone. The cost of rice husk varies from USD 100 to 180 for each metric ton from different vendors (Suhot *et al*., 2021). Proximate investigation of a husk test gives around 75 % unstable natural issue and the equalization of 25 % of the heaviness of this husk is ash and ordinarily known as rice husk debris (RHA). The major element of rice husk depends on silica (SiO_2) which is 92.20 wt. % (Hamdan *et al*., 2019).

Rice husk has free structure which is for the most part utilized for vitality creation. One of the most effective employments of this by product is its immediate combustion without requiring a heat exchanger with an appropriate heater to create heat for drying paddy. Consuming 1Kg husk gives approx. 3400 Kcal of vitality, which is about 1/3 of the vitality and produced from oil. However, the oil cost is lower multiple times in 2018, while gasification is the way toward changing over rice husk to a combination gas (syngas) in a gasifying reactor with a controlled air measure. As determined per Kg of rice husk can be 0.25 kW long periods of power and heat 4 kW h, contingent upon the innovation. Utilized structure materials are finely ground rice frames and can be blended in with different ingredients for example, coco peat, porous particles, concrete, added substances and glass fibre work. The weight of the block lighter materials, is around half and has sound proof which protected water proof properties. Rice bodies can be utilized as water filtration gear protecting hardware such as fillers and creating shiny metallic items and husk ash can be utilized as fertilizer (Arjmandi *et al*., 2017; Chen *et al*., 2016).

The thermal stability marginally expanded with the addition of the RH filler. Expanding RH's measure seems to influence the dispersion of fibre, bringing about agglomerating of the RH fibre and intermittence in the matrix. The penetrability coefficient esteems expanded with the joining and increment of RH in the hybrid filler. The examination has likewise indicated that the fuse of RH into LDPE expanded biodegradation altogether (Moura *et al*., 2017; Sam *et al*., 2017; Sharma 2017; Arjmandi *et al*., 2015; Chen *et al*., 2014). The examination has additionally indicated that the consolidation of RH into LDPE expanded biodegradation essentially which is a favourable position from an ecological perspective. The after effects of mechanical testing show that RH expansion in the hybrid composite, malleable, and tear quality decline while the tensile modulus increments. (Arabani and Tahami 2017; Moura *et al*., 2017). The thermal stability of reused thermoplastic mix rice husk (RH) improved with the expansion of (RH). RH filled LDPE composites have been found to better by and large mechanical properties contrasted with different composites for example, PLA, PP and PVC. The distinctive arrangement strategies have brought about composites with various mechanical, physical, warm and morphological properties. The limit modulus of bio composites was unimaginably overhauled by rice husk (Sam *et al*., 2017; Sharma, 2017). In this research, the effect of increasing rice husk content was tested on the mechanical and thermal properties of LDPE.

Materials and Methods

Two key materials are required to prepare the sample namely, LDPE and rice husk. LDPE and different reinforcement as prescribed by the manufacturing standards. Sigma Aldrich, Germany has purchased the LDPE. The product grade is 428043, density 0.925 g/mL at 25 °C and low density average Mw ~847. Rice husk was grinded in powdered form by using the grinding machine. It is taken in the form of refined powder form sized particles (≥ 60um). It is some what bigger than a rice grain, along these lines the lengths upto 7 mm are conceivable. Typical dimensions are 4 mm by 6 mm. Rice husk constitutes about 20 wt. % of rice and its composition is cellulose 50 wt. %, lignin 25–30 wt. %, silica 15–20 wt. % and moisture upto 10–15 wt.%. The rice husk has a low bulk density of around 90–150 Kg/m^3. The tests on reinforcement were performed to determine their physical properties.

The preparation of samples from rice husk reinforced LDPE involved four basic steps. The first step was to mix the rice husk in LDPE. The mixing was done in two roll mills. A100g sample was prepared with combined proportions of LDPE and rice husk. Appropriate mixing was done on two roll mills by keeping the temperature of the roller at 120°C. The mixed material is then placed in a mold sheet on a hot press. The temperature of the press is 160°C and the pressure is 200 psi. The samples are made to determine the thermal and mechanical properties of LDPE reinforced rice husk. Samples were prepared by using of four levels (1%, 2%, 5% and 10%) of substitution of rice husk with LDPE and pure LDPE conventional samples. The amount of rice husk was added by weight percent in samples and concerning the weight of reinforcement, that is 1% means 1g of reinforcement with 99g of LDPE. The samples were tested under the set conditions and From the results, dispersion and mechanical properties were obtained that characterize LDPE's behaviour concerning different fractions of rice husk reinforced.

The major components that have been used in the manufacturing of the sample which were LDPE and rice husk. Different weight proportions of both materials take part in the sample preparation with their diverse property. Both the materials are economically suitable because rice husk is obtained from the rice industry's waste and the other material, LDPE is a commercially available polymer.

Selection of equipment and development of sheet. The equipment is selected for its easy and efficient working at a faster production rate and a lower cost compared to other techniques available. The most effective thing is that in this, mixing and dispersion of material are proper.

Two roll mixing. The "two roll mill" with model number "LRMR-SC-110-W"was provided by Lab Tech Engineering Co. Ltd.In the two rolls mixing machine, the LDPE was added in granular form. The hot Rollers (at 100-120°C approx.) causes the LDPE to melt. The rice husk flour is added gradually to the melted LDPE. After rice husk had been mixed properly with the LDPE, a sheet is formed on the rollers. The thickness of the sheet depends on the nip gap and the size of the rollers. After mixing, the raw sheet is then moved to a hot press to be converted into the desired shape for further testing and characterization.

Hot press technique.A hot press machine is used to make samples for testing and characterization. The reinforced material from two roll mill is then placed in the mold. The pressure of 200psi (approx.) is applied at the temperature of 160°C approx. Under these conditions, the long chains break into smaller chains and the chains are aligned properly and give the sample a proper shape. The sample was heated for 5 min (approx.) and then cooled. The finished product is obtained and the strategy utilizes a split mold mounted in a hydraulic press. Figure 2 shows the pure and reinforced sheets of LDPE, LDPE with one wt. % rice husk and LDPE with five wt. % of rice husk.

UTM materials testing machine. With this equipment, the mechanical behaviour of the given sample can be determined by applying tensile, compressive and transverse stresses.

Tensile testing. The analysis of tensile tests is utilized to choose materials for designing applications. Tensile properties are often comprised of specifications of material to make certain quality. Tensile properties are frequently used to fore cast the performance of new materials and processes under sorts of materials and processes can be contrasted. The property of the material can be assessed by the stress that is required to cause obvious plastic distortion. The tensile test can determine the ductility and brittleness of the material. Tensile tests provide comprehensive information about yield strength, elongation, ultimate tensile strength and rupture.

Tear test. The tear test determines the force required to propagate a tear in a plastic sheet and film by the single tear method. The test is not favourable for brittle material. In the tear test, the stress is applied to material that contains partial rupture or fracture to carry the material to complete failure. The area on one side of the fracture is pulled up and the segment on the opposite side is pulled down. A consistent burden is then applied until the tear has spread the whole sample and the two areas have been separated from each other.

Samples are cut to a firmly characterized shape in a perfect world utilizing a die cutter. The cut samples are put under strain in a test stand set at a steady pace of measure, while the resultant tensile power is noted. The most extreme power in newton's and measures of prolongation in mm are accounted for. The test report will contain all the data important for the total proof of the sample. The average estimation of tear obstruction was determined in Newton's and the most extreme augmentation in mm, in addition to standard deviations of these averaged values.

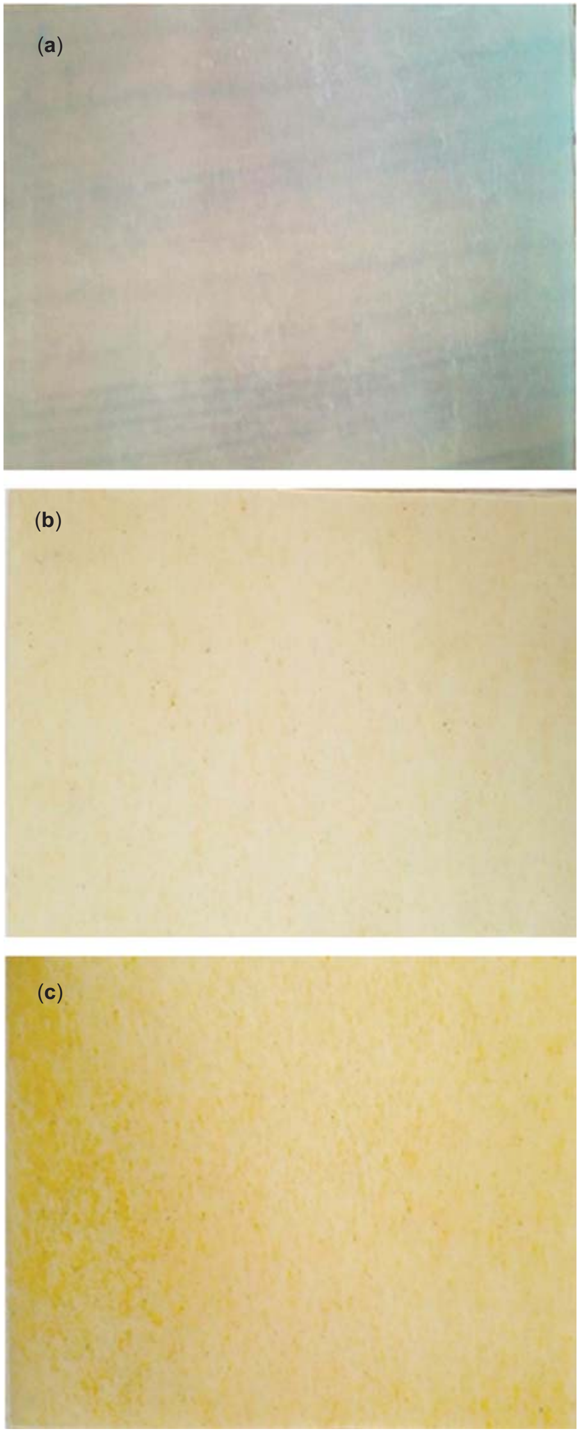

Fig. 2. Pure and blended sheets (**a**) Pure LDPE (**b**) LDPE withone wt. % Rice husk (**c**) LDPE withfive wt. % Rice husk

Thermogravimetric analysis (TGA). The Perkin Elmer Thermogravimetric analyzer was used to control the sample's change in weight concerning time and temperature. With the help of a thermal analyzer, the material's thermal strength can be measured along with used precursors. The analyzer can work under dynamic heating rates or isothermal conditions. A thermal analyzer can help check the volatile content, composition analysis and decomposition kinetics.

Fourier transform infra-red spectroscopy. Infrared spectroscopy is used to study the structural changes of the organic compound. FTIR provides valuable information concerning chemical changes. This information is very difficult to find in any other characterization techniques. The latest development in FTIR reduces the measurement time from minutes to seconds. Infrared Spectroscopy uses light between 4000 – 400/cm. The IR is changed into rotational energy, bending or vibrational energy when the molecule immerses in the light. The finger print area and the functional group area give information related to molecule and their functional group.

Perkin Elmer Spectrum one FTIR Spectrometer was utilized to distinguish a functional group's occurrence in a molecule by noting the particular absorption of radiation by every particle in the sample. FTIR analysis of the developed blend sheet examined the interaction between LDPE and rice husk. The scans were 20 with a wavelength range of 4000/cm – 450/cm and the spectra were obtained from a 200 micrometer diameter sampling area.

Hardness test. Hardness is the quality of anything or material that resists indentation, plastic distortion and scratching. These tests are empirically based on the experiment and observation. Strong intermolecular bonds usually describe these tests. The shore hardness test is commonly used for plastics. The shore hardness test is further classified as shore A and shore D both shore A for our composite. Itwas observed that the hardness value of our composite increases when rice husk content increases.

Results and Discussion

Characterization of development reinforced sheet. Different characterization techniques were used to evaluate the mechanical and thermal properties of the risk husk reinforced LDPE sheet. Mechanical testing involves TGA does the tensile test, tear test, hardness and thermal testing.

Tensile testing. Figures 3 and 4 show the results tensile test and modulus of rice husk reinforced LDPE.

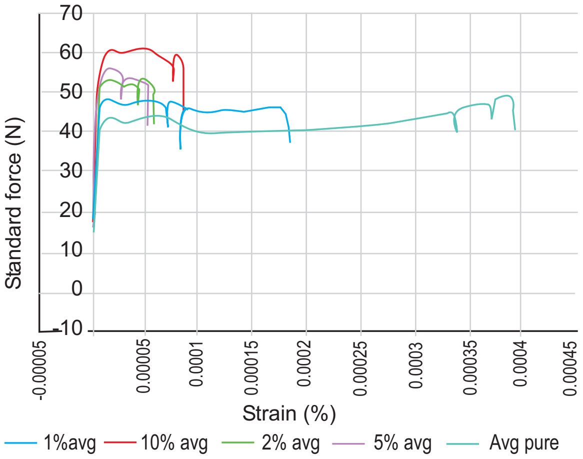

Fig. 3. Effect of rice husk reinforcement on tensile properties of LDPE.

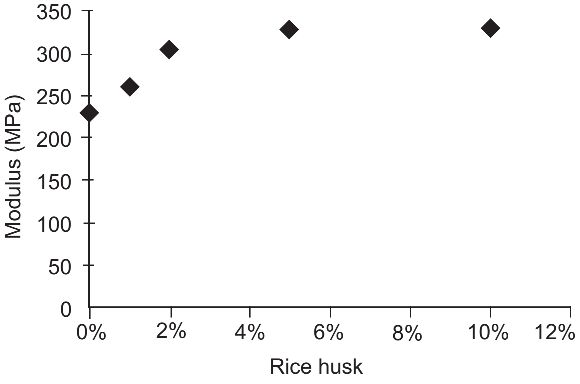

Fig. 4. Effect of rice husk reinforcement on LDPE.

According to the results shown, it was observed that by increasing the risk husk content, the force required to break the sample was also increased. For pure LDPE, the ultimate strength was observed at the force of 42N (approx.)with a strain of 3.9%.

After adding one wt. % of rice husk, the force was increased to 48N and the strain was 0.018%. For 2 %, 5 % and 10% reinforcement samples, the cross sectional area's force is 53N, 56N and 61N. The strain is approximately 0.0065%, 0.006% and 0.008%, respectively. It is observed the same thing. According to the results, rice husk has good compatibility with LDPE,so the increased percentage of rice husk has increased the ultimate tensile strength and modulus with a significant decrease in elongation strain. (Jacob *et al.*, 2019; Rajendran Royan *et al.*, 2018; Majeed *et al.*, 2014). Table 1 shows the brief results obtained from tensile testing.

Table 1. Pure LDPE and increases wt. % of rice husk by UTM

	Sample	E_t MPa	σM/mm^2	ε_M %	σ_B MPa	ε_B %	W_B Nmm	Hmm	Bmm	$A_o$mm^2
Pure LDPE	1	319.425	10.1262	337.6222	6.452503	343.0721	7025.092	1	5.9	4.425
	2	203.212	11.00712	387.3485	9.216565	393.7986	8381.814	1	5.9	4.425
	3	169.703	10.53127	365.9379	8.68097	373.1367	7699.017	1	5.9	4.425
	Avg.	230.720	10.55486	363.6362	8.116679	370.0025	7701.975	1	5.9	4.425
RH 1%	1	240.102	8.250528	50.12937	6.267968	183.9288	4237.187	1	6	6
	2	273.786	8.061509	53.16947	4.16457	82.66867	1888.936	1	6	6
	3	269.312	7.672337	14.63065	4.894448	69.58141	1504.562	1	6	6
	Avg.	261.067	7.994791	39.30983	5.108995	112.0596	2543.562	1	6	6
RH 2%	1	310.397	8.751618	15.31615	7.235196	43.69938	1053.401	1	6	6
	2	317.222	8.966408	13.11852	6.201829	42.13622	1025.746	1	6	6
	3	298.113	8.882214	45.6308	6.969596	57.88009	1438.827	1	6	6
	Avg.	308.577	8.866746	24.68849	6.802207	47.90523	1172.658	1	6	6
RH 5%	1	321.779	9.069578	14.21223	6.90102	52.82935	1335.813	1	6	6
	2	336.422	9.478558	15.64058	7.203674	27.76524	700.569	1	6	6
	3	336.312	9.211354	15.09823	7.00231	27.60120	980.4071	1	6	6
	Avg.	329.101	9.274068	14.92641	7.052347	40.29729	1018.191	1	6	6
RH 10%	1	346.167	10.33169	52.05482	7.597655	86.63072	2539.121	1	6	6
	2	339.966	10.59874	48.3308	7.777765	76.4808	2284.577	1	6	6
	3	307.477	9.44655	47.35366	7.10455	74.23685	1953.897	1	6	6
	Avg.	331.203	10.12566	49.24643	7.493323	79.11612	2259.198	1	6	6

Tear test result. Figure 5 shows that as % of reinforcement increases, tear growth resistance (R) decreases. The density of the material is increased as ricehusk is denser than LDPE. Tear force also decreases as % of reinforcement increases. (Barczewski *et al*., 2017; Rassiah *et al*., 2016) Tear resistance might also be decreased because of stress concentration areas present in the areas where the force would act.

Result of TGA. The material's thermal stability increased upon the reinforcement with rice husk, as rice husk has higher thermal stability than LDPE as shown in Figure 6. As rice husk particles are dispersed within the LDPE matrix, they tend to increase the thermal stability by enduring more heat (Zhang *et al*. 2018; Zhang *et al*. 2017).

Fourier transform infra-red spectroscopy. FTIR is an advantageous technique to distinguish primary classes of polyolefin films. A definite interpretation of the infrared range will empower even fundamentally the same as structures to be recognized.

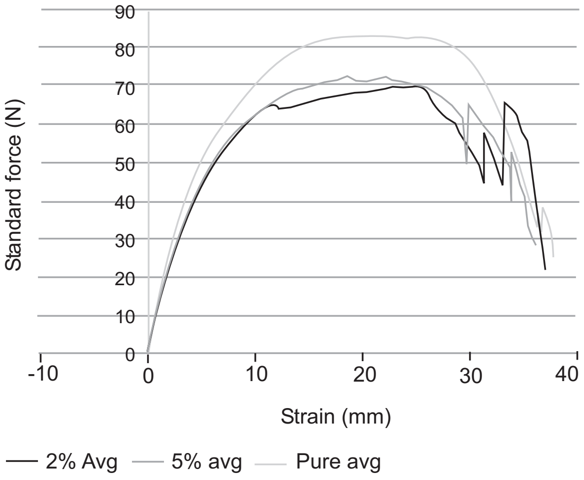

Fig. 5. Effect of tear test on adding the different composition of rice husk in LDPE.

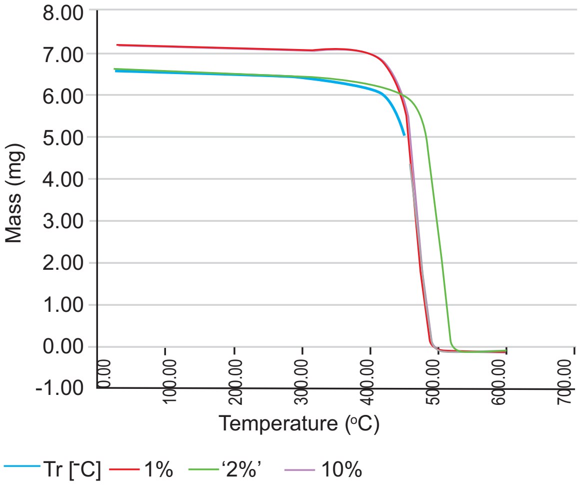

Fig. 6. Effect of TGA on adding the different composition of rice husk in LDPE.

The degree of expansion can be estimated and crystallinity can be indirectly estimated. According to the results of Fig. 7, the fourier transform infrared spectroscopy indicates that there is no change in the chemical properties of the matrix, the composite formed is just a physical mixing of rice husk with LDPE. The radiation graphs are the same for both pure and modified LDPE (Zhang *et al*., 2019; Chen *et al*., 2016).

Hardness test result. Figure 8 shows that, by increasing the % of the reinforced particle, the hardness of the composite increases. When 2%, 3%, 5% and 10% rice husk is incorporated, the sample shows 68, 70, 72 and 80 Shore A, respectively.

By increasing the % of the reinforced particle, the hardness of the composite increases. The future recommendation using this particulate composite as a matrix for fibre reinforced composites or sandwich panels, modify the material for other molding techniques, combining some other particles with rice husk in LDPE compatibilizer, or using surface treated rice husk. (Arjmandi *et al*., 2015; Chen *et al*., 2015; Chen *et al*., 2014; Majeed *et al*., 2014). Hence, adding rice husk in LDPE shows good results and increases the thermal and mechanical properties. The results obtained are in good agreement with the previous literature (Alim *et al*., 2021; Azam *et al*., 2020).

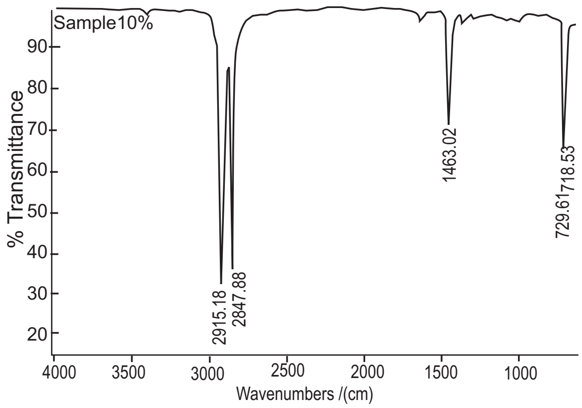

Fig. 7. FTIR spectra of LDPE with rice husk.

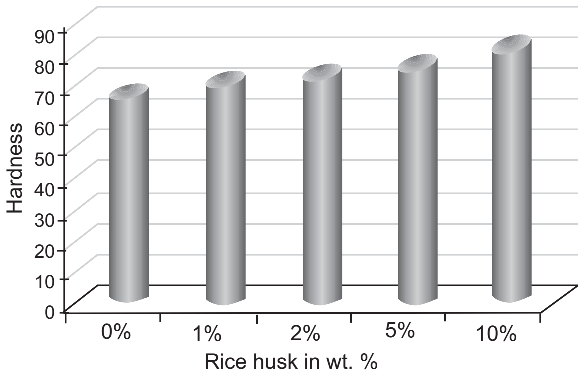

Fig. 8. Hardness versus % reinforcement of pure LDPE and LDPE with rice husk

Conclusion

Today, industries compete to provide the requirement in the minimum budget. In this way, particulate composites are becoming more popular day by day. They provide better properties especially excessive strength due to interfacial tension, pertinent to many applications at low cost. The LDPE is selected because of its reasonable characteristics, easy processing, and moderate properties, while the rice husk is light weight and cost effective. Tensile and thermal testing was executed on the specimen, prepared using compression molding for which results indicated better performance. The research results show that the ultimate tensile strength, modulus, thermal resistance and hardness were increased with the increasing content of rice husk as the rice husk particles showed good dispersion with LDPE. On the other side, the elongation and tear resistance is decreased because increasing rice husk particles increased the stiffness of the composite. According to the results of the fourier transform, infrared spectroscopy indicates that there is no change in the chemical properties of the matrix. The composite formed is just a physical mixing of rice husk with LDPE. Hence, rice husk was observed to have improved the countenances of the material leading to better performance and relatively cost effective production.

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Conflicts Of Interest. The authors declare that they have no conflict of interest.

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