



CHARACTERIZATION OF RECYCLED LOW DENSITY POLYETHYLENE AND EGGSHELL PARTICULATE COMPOSITE (RLDPE/ESP)

*Abdulrahman, A. S. and Haruna, V. N.

Department of Mechanical Engineering, Federal University of Technology, Minna, Niger State, Nigeria

ARTICLE INFO

Article History:

Received 25th January, 2015
Received in revised form
02nd February, 2015
Accepted 16th March, 2015
Published online 30th April, 2015

Key words:

Particle-reinforcement, Environmental degradation, Mechanical testing, Recycling.

ABSTRACT

This paper focused on the effects of ESP loading on the properties and microstructure of RLDPE and ESP composite. Polymer composite samples were fabricated by compounding the composites using two roll mill and compressing them in the mould using hydraulic press. 5 to 25 wt% ESP of size 75µm were added at intervals of 5 wt% to the polymer as reinforcement. The results showed that the tensile strength increases to a maximum value of 19.43 MPa at 10 wt% ESP; the Young's modulus has a maximum value of 100 MPa at 20 wt% filler content; The density of the composite increases with increase in filler loading while the water absorption rate increases with time of exposure and the flame propagation rate decreases with increase in filler content. Hence the achieved composite will be relevant in civil, electronics and automobile industries.

Copyright © 2015 Abdulrahman, A. S. and Haruna, V. N. This is an open access article distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

INTRODUCTION

The modern development in the field of science and technology demands the developments of advanced engineering materials for various engineering applications, especially in the field of transportation, aerospace and military engineering related areas. These areas require light weight, high strength, and high stiffness having good tribological properties (Jokhio *et al.*, 2011). Such demands can best be met only by developing and processing composite materials. Composites are materials obtained by combining two or more physically and/or chemically distinct phase, suitably arranged or distributed and having characteristic features that are not exhibited by any of its components in isolation (Al-Mosawi *et al.*, 2012). Composite materials consist of a reinforcement or filler usually in fibres/particles form embedded within a matrix, a material that surrounds and tightly bound to the fibres. The matrix may be metallic, ceramic or polymeric in origin. It gives the composites their shape, surface appearance, environmental tolerance and overall durability while the fibrous reinforcement carries most of the structural loads thus giving macroscopic stiffness and strength (Encyclopaedia of Polymer Science Engineering, 1985). The most widely used type of composite material is Polymer Matrix Composites (PMCs). Polymer composites can be fabricated into different shapes using known polymer melt processing parameters, such as films, wires and bulk materials which is dependent on the rheological melt behaviour of the polymer (Tahar *et al.*, 1997; Capozzi *et al.*,

2004; Gupta *et al.*, 2006; Baughman *et al.*, 2002; Kota *et al.*, 2007; Greco and Maffezzoli, 2003; Haruna *et al.*, 2014). A composite material can provide superior and unique mechanical and physical properties because it combines the most desirable properties of its constituents while suppressing their least desirable properties. The opportunity to develop superior products for aerospace, automotive, and recreational applications has sustained the interest in advanced composites (Schuh, 2000; Eder *et al.*, 2006). Therefore, this investigation focused on the effect of ESP loading on the properties and microstructures of RLDPE and ESP composite since there is presently a perceived shortage of wood fibre for producing composites as a result of fibre competition by pulp mills, reduced harvesting and manufacturing and diminished log quality while chicken ES that has been listed worldwide as one of the worst environmental problems and RLDPE are available wastes in abundance that can be used as substitute for sustainability of polymer composites production.

MATERIALS AND METHODS

Materials

RLDPE and ES were collected as waste materials from dump site. NaOH pellets and distilled water were purchased from Nahson Nigeria Limited, 309 AA Hospital Road, Minna, Niger State and Department of Science Laboratory Technology, Federal Polytechnic, Bida, Niger State respectively. Foil paper used as lining on the inner surfaces of the moulds during casting for easy removal of the cast composites was purchased

*Corresponding author: Abdulrahman, A. S.
Department of Mechanical Engineering, Federal University of Technology, Minna, Niger State, Nigeria.

in a shop at the gate of National Institute of Leather and Science Technology (NILEST), Samaru, Zaria, Kaduna State.

Equipments

The equipments used in this research work were: Two roll mill for compounding the composites; hydraulic press for compressing the composites; Charpy Impact Testing Machine; Tensometer; Enerpac Universal Materials Testing machine and Shore A Hardness Tester for impact, tensile, flexural and hardness tests respectively. However, digital weighing machine was also used for weighing the RLDPE, ESP and the fabricated composites.

Preparation of the Eggshell Particulate

The eggshells (ES) collected were soaked in water for two (2) hours for easy removal of the membrane from the inner surface of the shells. The ES were then cleaned properly with water to remove contaminants and eliminate odour and dried under sun for three and a half (3½) hours before grinding it to powder using grinding machine. The resulting powder was sieved to obtain an average particle size of 75µm. This is in accordance with the method adopted by Nwanonyi and Chike-Onyegbula, 2013.

Treatment of the ES Particulate

The ES powder was chemically treated with 10% NaOH solution in accordance with the procedure adopted by Shuhadah and Supri, 2009. The powder was mixed with this solution and stirred properly for five (5) minutes and kept at room temperature until two layers are formed. The upper layer was removed and the bottom layer was washed several times with distilled water to remove the presence of NaOH from it. The resulting precipitate was dried in the oven at 40°C for some hours until it was observed that its weight remains constant.

Compounding of the Composites

The composite samples were compounded using Two Roll Mill model 5183 at a temperature of 150°C to allow for adequate flow of the molten polymer (RLDPE) before pouring the ES powder which is used as filler for the composite. Cross mixing of the molten matrix and the filler was done properly for 10 minutes to obtain homogeneity.

The band of the composites formed round the front roll of the mill was removed after compounding using heat resisting hand gloves and placed in metallic mould and then compressed to the required thickness by an electrically heated hydraulic press (Greco and Maffezzoli, 2003; Hassan *et al.*, 2012).

Foil paper was used to line the inner surface of the mould to allow for easy removal of the composite after curing. The composite was finally removed from the press for cooling at the end of the press cycle. Six different types of composites were casted in accordance with the formulation shown in Table 1.

Table 1. Formulation of the Composites

Sample	RLDPE (wt %)	ESP (wt %)
1	100	0
2	95	5
3	90	10
4	85	15
5	80	20
6	75	25

Characterization and Measurements

The tensile test was conducted on the Monsanto Tensometer Tensile Testing Machine. The specimens were cut to a dimension of 120 × 15 × 5mm with a gauge length of 40mm marked using a scribe. Three specimens were tested for each of the samples of the composite and the maximum force was recorded and used for tensile strength determination according to ASTM, 2000. Flexural test was also carried out on a 100KN Enerpac Universal Materials Testing machine model Cat. Nr. 261. The specimens of the samples were cut to a dimension of 100 × 50 × 5mm and bent using an improvised support and a centre point load to breaking point (Casaurang *et al.*, 1991; Jokhio *et al.*, 2011). The impact test was carried out using 15 J & 25 J Capacity Charpy Impact Testing Machine with model number Cat. Nr. 41-07-15269C. The sample specimens were cut to a dimension of 75 × 10 × 10mm using hack saw. A 45° V-notch of 3mm deep was then cut on one of the faces which provide stress concentration during the test. The sample was then placed on the machine and the pendulum was raised and allowed to swing-fall under gravity to hit the specimen (ASTM, 2000). Hardness values of the composites were determined using Shore A Durometer hardness testing instrument in accordance with ASTM D2240. The geometry of the specimens is 100 × 50 × 5mm. The term durometer is often used to refer to the measurement as well as the instrument itself. Durometer is typically used as a measure of hardness in polymers, elastomers, and rubbers (Kota *et al.*, 2007). The densities of the composite samples were determined using Eq. (1) (Jokhio *et al.*, 2011):

$$\rho = \frac{m}{v} \quad (\text{g/cm}^3) \quad \dots\dots\dots (1)$$

Where: ρ = Density (g/cm³), m = Mass (g) and v = Volume (cm³).

The samples are in square shapes having their dimensions as 20 × 20 × 10mm. The volumes were calculated using the parameters of the samples that are, by multiplying the length, width and the thickness together (lwt). However, the masses were measured using a digital weighing machine. The water absorption test of the samples was carried out in accordance with ASTM standard D750-95. The samples were properly labelled in accordance with their formulation and weighed with digital weighing machine to get their initial masses before they were fully immersed into the water in a container at room temperature in a laboratory. The masses of the samples were measured at interval of 96 hours for a total of 384 hours to obtain the amount of water absorbed. Water absorption rate (M_t) was obtained using Eq. (2) (Kozlowski and Helwig, 1998):

$$M_t = \frac{(W_n - W_d)}{W_d} \times 100\% \quad \dots\dots\dots (2)$$

Where W_d and W_n are the dry and wet weights of the samples after exposure respectively. The samples for this test are of same dimensions as the samples for the densities determination. Flammability test was also carried out. In this work, a 50mm mark was measured and marked out on the concerned samples. The samples were then clamped horizontally in a bench vice with the 50mm mark protruding out of the vice. The protruding ends were ignited and the times taken for the samples to ignite were recorded as the ignition time (I_t). The samples were allowed to burn to 50 mm mark (D_p). The relative burning rate of individual sample was obtained using Eq. (3) (Ewulonu, C. M. 2009. M. Sc. Thesis, FUTO, Nigeria):

$$P_r = \frac{D_p}{(P_t - I_t)} \text{ (mm/s)} \quad \dots\dots\dots (3)$$

Where: P_r = Flame propagation rate (mm/s), D_p = Propagation distance (mm), P_t = Flame propagation time (seconds) and I_t = Ignition time (seconds). The morphological structure and the chemical compositions of the phases present in the test samples were studied using PHENOM Prox Scanning Electron Microscope (SEM) model JOEL JSM-6460LA.

RESULTS AND DISCUSSION

The result of tensile strength is as shown in Fig. 1. It can be observed that the tensile strength of the composite increases from 13MPa to a maximum of 19.43MPa at 0 and 10 wt% ESP respectively and then decreased steadily to 13.17MPa at 25 wt% ESP. The increase in the tensile strength may be attributed to the better surface area of the particles in the matrix after chemical treatment. Increased in interfacial area gives rise to good interfacial bonding between the hydrophilic particles and hydrophobic matrix. These were similarly observed in the work (Mishra *et al.*, 1999; Mix and Giacomini, 2011; Mohanty *et al.*, 2005; Schuh, 2000; Shuhadah *et al.*, 2008; Tahar *et al.*, 1997; White and Ansell, 1993).

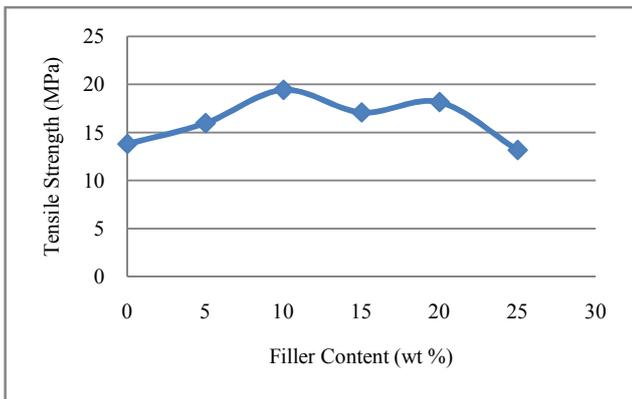


Fig 1. Variation of Tensile Strength with Filler Content

Variation of the Young’s Modulus as a function of filler content is as depicted in Fig. 2. The values of the Young’s modulus investigated range from 61.9 MPa to 100 MPa at 25 wt% and 20 wt% respectively. Variation of the percentage

elongation with filler content is shown in Fig. 3. It can be seen that the elongation at break of the composite decreases with increasing filler loading except at 10 wt% and 25 wt% where sudden rises can be seen. These sudden rises may be attributed to the lower extent of the homogeneity of the mixture before casting the composite. However, the reduction in the elongation at break with increasing filler loading indicates the incapability of the filler to support the stress transfer from filler to matrix.

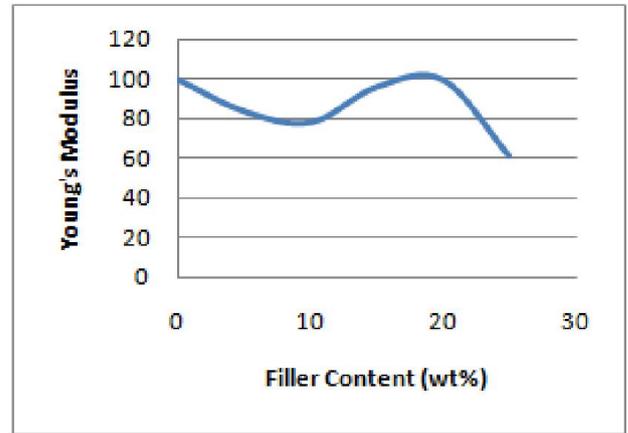


Fig 2. Variation of Young’s Modulus with Filler Content

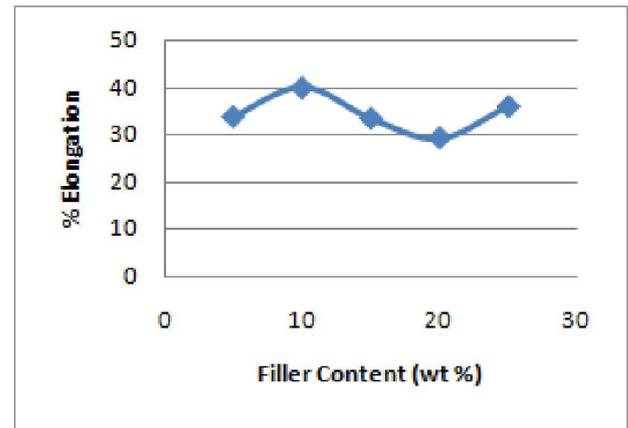


Fig 3. Variation of % Elongation with Filler Content

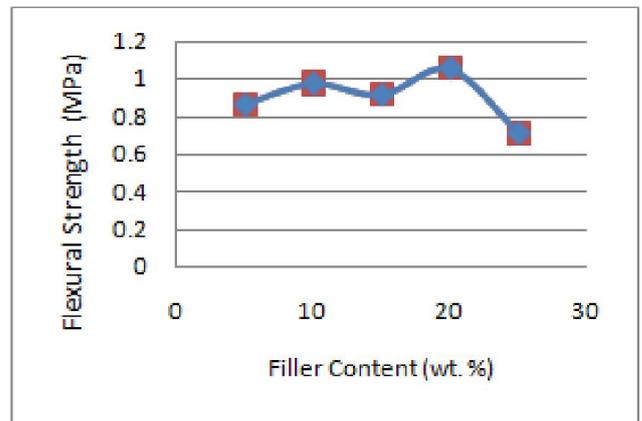


Fig 4. Variation of Flexural Strength with Filler Content

Figure 4 shows the variation of flexural strength with filler content. The flexural strength increases to 0.98MPa at 10 wt% of filler content and then decreases to 0.92MPa at 15 wt% filler content before increasing again to 1.06MPa at 20 wt% filler content and finally falling down to 0.72MPa at 25 wt% filler content. The decrease in flexural strength after the maximum value is attributed to weak interfacial bonding as the wt% filler particles increases to 25 wt% which resulted to weak the bonding load carrying by the matrix (Mishra *et al.*, 1999; White and Ansell, 1993). Variation of flexural modulus with filler content is displayed in Fig. 5 and behaviour is in a similar manner as that of Fig. 4.

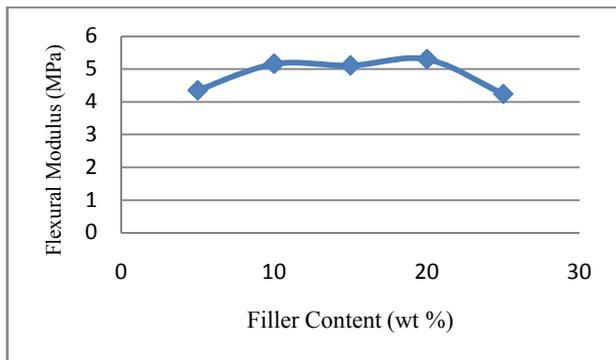


Fig 4. Variation of Flexural Strength with Filler Content

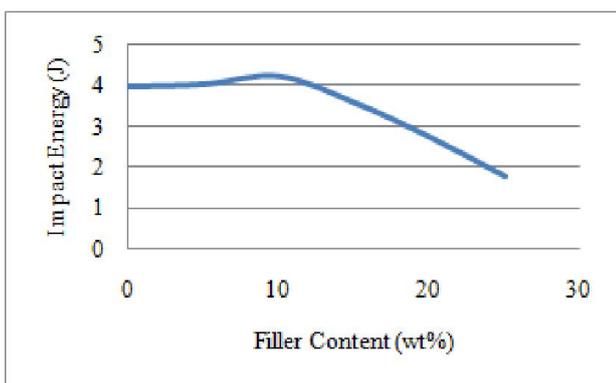


Fig 6. Variation of Impact Energy with Filler Content

Variation of impact energy with filler content can be observed in Fig. 6. The impact energy of the composites increases to 4.25J at 10 wt% filler content and then steadily decreases to 1.78J at 25 wt% filler content.

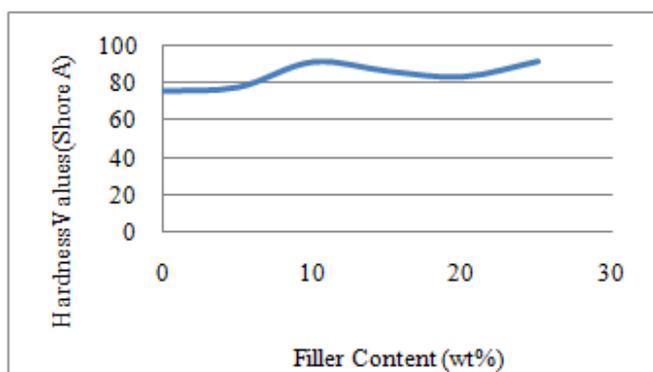


Fig 7. Variation of Hardness Values with Filler Content

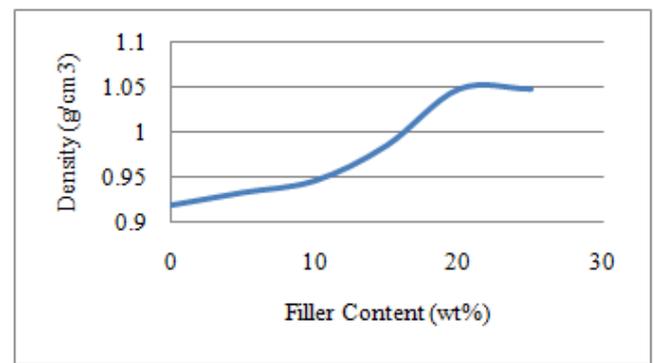


Fig 8. Density Variation with Filler Content

Variation of the hardness values of the composite with filler content is exhibited in Fig. 7. The hardness value ranges from 76 at 0 wt% filler content to 92 at 25 wt% filler content. However, the increase in filler content increases the hardness and stiffness of the composite. It can be observed from Fig. 8 that the density of the RLDPE reinforced with ESP composites increases with increase in the filler content. This work is in line with the earlier work carried out by Hassan *et al.*, 2012 and Chira, C. V. 2014. M. Eng Thesis, ABU, Zaria, Nigeria.

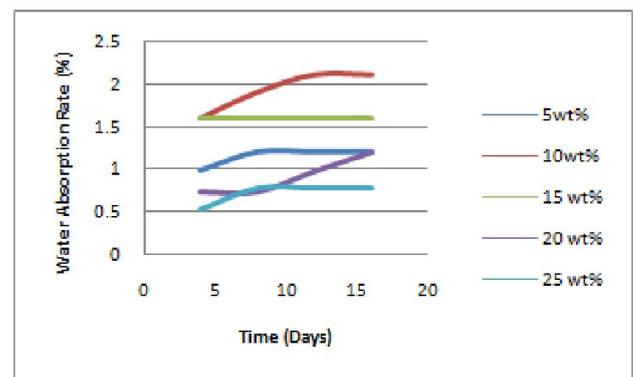


Fig 9. Water Absorption Rate Variation with Time

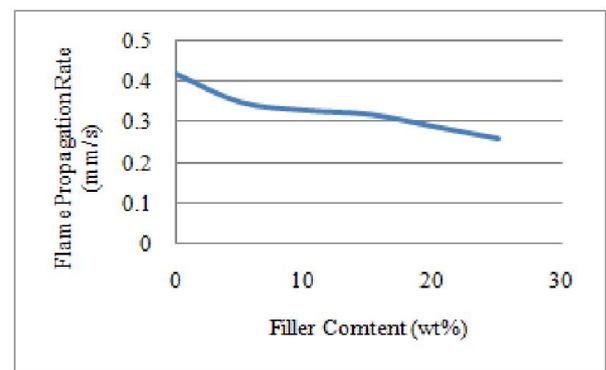


Fig 10. Flame Propagation Rate Variation with Filler Content

Variation of water absorption rate of the composite as a function of time exposure with different filler content is shown in Fig. 9. This experiment was carried out over a period of 384 hours (16 days) and the samples were weighed at intervals of every 96 hours (4 days). In almost all the filler loadings, there was increase in water absorption rate in the first 188 hours except for the composite with 15wt% ESP which represents the

most stable sample, where the absorption rate was constant throughout. Other filler loadings have stable absorption rate after 288 hours except for composite with 20wt% ES particle where the absorption rate was increasing throughout the period of the experiment. However, the overall result shows that the water absorption rate increases with increase in time of exposure. This is similar to the report of Nwanonenyi and Chike-Onyegbula, 2013. Variation of flame propagation rate with filler content is shown in Fig. 10. It can be observed that the flame propagation rate of the composite decreases steadily with increasing filler content as expected because of the nature and chemical composition of the eggshell. This work is in line with the earlier work carried out by Nwanonenyi and Chike-Onyegbula, 2013. The SEM/EDS microstructures of the composites are shown in Figs 11-15

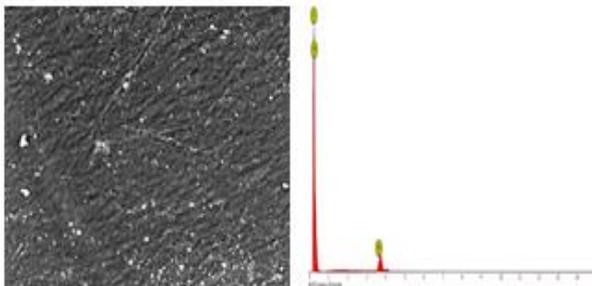


Fig 11. SEM/EDS microstructure of 5 wt% ESP in RLDPE

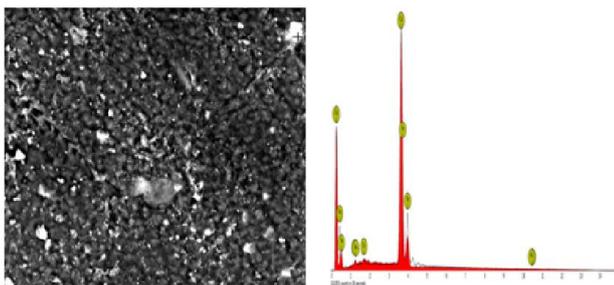


Fig 12. SEM/EDS microstructure of 10 wt% ESP in RLDPE

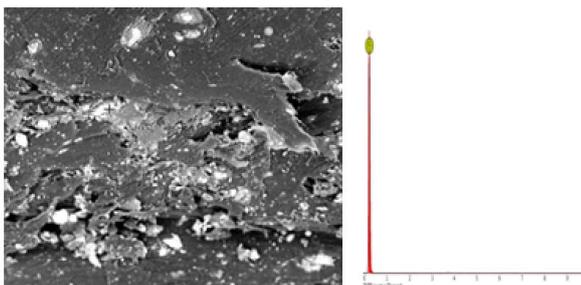


Fig 13. SEM/EDS microstructure of 15 wt% ESP in RLDPE

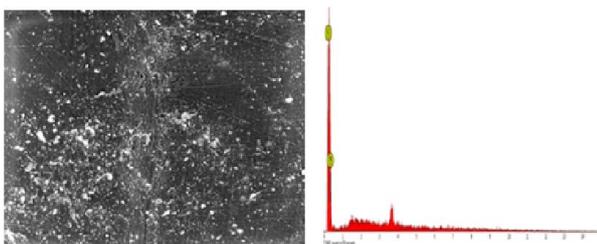


Fig 14. SEM/EDS microstructure of 20 wt% ESP in RLDPE

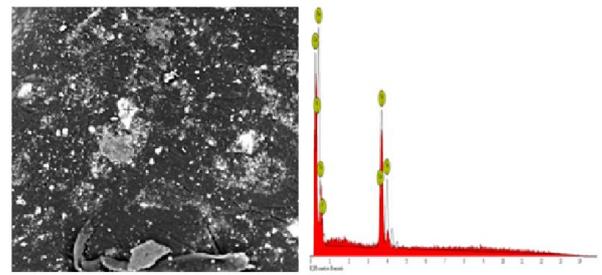


Fig 15. SEM/EDS microstructure of 25 wt% ESP in RLDPE

Conclusion

From the results of the investigation, the following conclusions can be drawn:

- RLDPE/ES particulate composites were successfully fabricated by compounding and compression moulding.
- The cast composite has the maximum value of tensile strength of 19.45 MPa at 10 wt% ESP indicating a better surface area of the particles in the matrix at this loading after chemical treatment.
- The percentage elongation of the composites decreases with increasing filler loading indicating the incapability of the reinforcement to support the stress transfer from filler to matrix.
- The decrease in flexural strength after the maximum value is attributed to weak interfacial bonding as the wt% of the filler particles increases.
- The impact energy of the cast composites decreases as the filler content increases except at 10 wt% of filler loading where there was an initial rise.
- The hardness of the composites assumes a sinusoidal waveform as the filler loading increases. This shows that the increase in filler loading causes an increase in the hardness of the composites for a short while.
- The density of the composites increases with increase in filler loading.
- Good interfacial bonding between the ESP and the RLDPE gives rise to low water absorption rate.
- The flammability of the composites decreases as the filler loading increases.

Acknowledgements

The authors would like to express their gratitude to Polymer Processing Unit of National Institute of Leather and Science Technology, Zaria and Mechanical Engineering, Metallurgical Engineering, and Chemical Engineering Departments of Ahmadu Bello University, Zaria for compounding the composites and providing the necessary equipments to carry out all the tests and SEM/EDS analysis respectively.

REFERENCES

- Al-Mosawi, A. I., Ammash, H. K. and Salaman, A. J. 2012. Properties of Composite Materials Databook. 2nd edition, Lambert Academic Publishing LAP.
- ASTM D 638-99-2000 and 790-99-2000. ASTM Committee on Standards, American Society for Testing and Material.

- Baughman, R. H., Zakhidov, A. A. and De Heer, W. A. 2002. Carbon Nanotubes: *The Route Toward Applications. Science.*, 297: 787-792.
- Capozzi, C.J., Shackelford, S, Ou R and Gerhardt, R.A. 2004. Study of Percolation in PMMA/Indium Tin Oxide Composites. Proceedings of the Symposium on Materials Research Society, Volume 819, Warrendale, PA., USA.
- Casaurang, M. Herrera, P. Gonzalez, I and Aguilar, V.M. 1991. Physical and Mechanical Properties of Henequen Fibres. *Journal of Applied Polymer Science.*, 43(4): 749-756.
- Eder A, Strobi S and Schwarzbuer P. 2006. Worldwide Market Report on Wood Plastic Composites. Wood K Plus. Encyclopaedia of Polymer Science Engineering, 1995.
- Greco, A, and Maffezzoli, A. 2003. Polymer Melting and Polymer Powder Sintering by Thermal Analysis, *J. Therm. Anal. Calorim.*
- Gupta, and Surappa, 2006. "Processing Microstructure Properties of Al Based MetalMatrix Composites", *Journal of Key to Engineering Materials.*, 104(107): 259-274.
- Haruna, V. N., Abdulrahman, A. S., Zubairu, P. T., Isezuo, L. O., Abdulrahman, M. A., and Onuoha, D. C. 2014 Prospects and Challenges of Composites in a Developing Country. *APRN Journal of Engineering and Applied Sciences.*, 9(7): 1069-1075
- Hassan, S.B.; Aigbbodion, V.S. and Patrick, S.N. 2012. Development of Polyester/Eggshell Particulate Composites. *Tribology in Industries.*, 34(4): 217-225.
- Jokhio, M. H.; Panhwar, M. I. and Unar, M. A. 2011. Manufacturing of Aluminium Composite Material Using Stir Casting Process. *Mehran University Research Journal of Engineering & Technology.*, 30(1): 53-64.
- Kota, A. K., Ciprano, B.H., Duesterberg, M. K., Gershon, A. L., Powell, D., Raghavan, S. R. and Bruck, H. K. 2007. Electrical and Rheological Percolation in Polystyrene/MWCNT Nano Composites. *Macromolecules.*, 40: 7400-7406.
- Kozlowski, R. and Helwig, M. 1998. Lingo Cellulosic Polymer Composite, Inc: Prasad P. N. (Ed.), Science and Technology of Polymers and Advanced Materials, Plenum Press, New York: pp 679-698.
- Mishra, S. C., Nayak, N.B. and Satapathy, A. 1999. Investigation on Bio-waste Reinforced Epoxy Composites. *Journal of Reinforced Plastics and Composites.*, 29(19): 3016-3020.
- Mix, A. W. and Giacomini, A. J. 2011. Standardized Polymer Durometry. *Journal of Testing and evaluation.*, 39 (4): 1-10.
- Mohanty, A.K. Misra, M. and Drzal, L.T. 2005. Natural fibres, Biopolymers and Biocomposites, *Taylor & Francis, New York.*
- Nwanonyi, S.C. and Chike-Onyegbula, C.O. 2013. Water Absorption, Flammability and Mechanical Properties of Linear Low Density Polyethylene/Eggshell Composite. *Academic Research International.*, 4(1): 352-358.
- Schuh T.G. 2000. Renewable Materials for Automotive Applications. Stuttgart: Daimler- chrysler AG.
- Shuhadah, S. and Supri, A.G. 2009. LDPE-Isophthalic Acid Modified Eggshell Powder Composites (LDPE/ESP₁). *Journal of Physical Sciences.*, 20(1): 87-98.
- Shuhadah, S., Supri, M. and Kamaruddin, H. 2008. Thermal Analysis, Water Absorption and Morphology Properties of Eggshell Powder Filled Low Density Polyethylene composites, in: Proceedings of MUCET, UniMAP, Kangar, Perlis: pp. 15-16.
- Tahar, R. B. H., Ban, T., Ohya, Y. And Takahashi, Y. 1997. Optical, Structural, and Electrical Properties of Indium Oxide Thin Films Prepared by the Sol-Gel Method. *Journal of Applied Physics.*, 82: 865-870.
- White, N. M., Ansell, M. P. 1993. Straw Reinforced Polyester Composites. *Journal of Materials Science.*, 18(5): 1549-1556.
