IOP Conf. Series: Materials Science and Engineering **149** (2016) 012043 doi:10.1088/1757-899X/149/1/012043

A bio-material: mechanical behaviour of LDPE-Al_2O_3-TiO_2

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Abstract. Polymer composites are prominent candidate for polymeric bio-composites due to its low cost, high strength and ease of manufacturing. However, they suffer from low mechanical properties such as high wear rate and low hardness. In view of this, present study focuses on the synthesis of hybrid bio polymer matrix composites using low density polyethylene as matrix material with reinforcing material namely, alumina and titanium oxide. The samples were fabricated as per ASTM standard by varying the percentage of reinforcing particles using injection moulding machine. Various tests namely, tensile, flexural, impact, hardness, wear, SEM and corrosion were conducted on the prepared samples. On the basis of the experimental results, it can be concluded that injection moulding process can fabricate defect free cast samples. Polymer matrix composites of 70%LDPE +10% TiO_2 +20% Al_2O_3 composition is biocompatible and a good candidate for biomaterial. Thus based on the inference of this study the above polymer matrix composite is suitable for orthopaedic applications and can be applied on hard and soft tissues of implantable materials in a human body.

1. Introduction

Recently, polymer metal or/and ceramics matrix composites are finding applications as biomaterial. It acts together in human tissue and body fluids to treat, improve, or replace anatomical elements of the human body. Biomaterials are used in medical devices for implants. An important factor that influences the progress of potential biomaterial is the mechanical properties of such materials.

Polyethylene is one such polymer material which is easily available, has relatively low cost and can be processed at 150-250^{0}C temperature. Polyethylene's are cross linked, and are of linear low density polyethylene (LLDPE), low density polyethylene (LDPE), high density polyethylene (HDPE). Branched structure is more suitable for cross linking. Therefore, cross linking of LDPE and HDPE requires more attention. Cross linked polyethylene forms a dense network of high molecular weight, which improves impact strength, environmental stress crack resistance, creep and abrasion resistance without influencing tensile strength and density to any appreciable extent [1].

Further to improve the wear properties of low density polyethylene alumina (Al_2O_3) combined with titanium oxides (TiO_2) are used by the researchers. Alumina is thermodynamically stable up to a temperature 2000^{0}C and it is a hard material with a hardness value of 1900HV. Also, alumina is available in both ionic bonds and covalent bonds. Titanium and its alloys are of particular interest for biomedical applications because of their outstanding biocompatibility, no reaction with tissue surrounding the implant, especially as hard tissue replacements as well as in cardiac and cardiovascular applications [2]. Also, a titanium oxide has the potential to stop the corrosion by reducing the electronic exchange process. Titanium oxide has isoelectric point which can be retained at pH (5-7) values of the human body. These materials are classified as biologically inert biomaterials. As such, they remain essentially unchanged when implanted into human bodies. The human body is able to recognize these materials as foreign and tries to isolate them by encasing it in fibrous tissues. However, they do not promote any adverse reactions and are tolerated well by the human tissues [3]. Following paragraphs illustrates the review of research work carried out on the mechanical properties of the bio-materials developed by the researcher's in the past. Thamaraiselvi and Rajeswari (2004) have developed alumina and zirconia ceramics as bio-ceramics due to their biocompatibility for implant. Results showed high mechanical strength with minimal or no tissue reaction, nontoxic to tissues and blood compatibility [4]. Thimmanagouda et al. (2014) studied mechanical properties of the 12%, 24% and 36% of hybrid fiber (natural fiber- Sisal, Jute and Hemp) polymer composite material. They compared the mechanical properties of new developed polymer composite materials with the femur bone strengths. Also it was found that by increasing the percentage of the fiber, the mechanical properties were increased. They found tensile strength of 12%, 24%, and 36% is having 0.096 kN/mm^2 0.093 kN/mm^2, 0.169k N/mm^2, compressive strength 0.102 kN/mm^2, 0.134 kN/mm^2, 0.140 kN/mm^2 and bending strength of 1.244 kN/mm^2, 1.313 kN/mm^2, 1.377 kN/mm^2 respectively. Later they compared these results to the femur bone tensile strength, compressive and bending strength $0.04344^{\pm 0.00362}$ kN/mm^2, $0.11529^{\pm 0.01294}$ kN/mm^2 and $0.084^{\pm 0.00991}$ kN/mm^2 respectively [5]. Dinesh (2013) investigated the tensile strength and compression strength of 10%, 20% and 30% natural (Sisal) fiber reinforcement epoxy composite materials. As per the study they found that the tensile and compressive strength of 30% natural (Sisal) fiber reinforcement epoxy composite material are 77 N/mm^2 and 64.66 N/mm^2 respectively [6]. Kumar et al. (2013) studied the mechanical properties of banana fiber, glass reinforced hybrid polypropylene composites (BGPP) by varying fiber weight percentages. The recorded tensile strength of pure polypropylene and banana fiber, glass reinforced hybrid polypropylene composites (BGPP) was 23.80 MPa and 24.59 MPa at 10% fiber fraction respectively. The recorded flexural strength of pure polypropylene and banana fiber, glass reinforced hybrid polypropylene composites (BGPP) was 227.57 MPa and 270.86 MPa at 10% fiber fraction respectively. Subsequently, the impact strength of pure polypropylene and banana fiber, glass reinforced hybrid polypropylene composites (BGPP) was 19.25 J/m and 29.37 J/m at 10 weight % fiber fraction respectively [7]. Rao et al. (2014) investigated the mean tensile strength, mean flexural strength, and mean impact strength of bamboo fiber filled with fly ash filler reinforced hybrid composites. They prepared the specimens by using hand lay-up technique and were tested as per ASTM standards. They found that the mean tensile strength of 84.61 MPa, mean flexural strength of 233.02 MPa and mean impact strength of 17.02 kJ/m [8]. Olaitan et al. (2014) studied morphology and mechanical properties of coconut shell reinforced with epoxy resin composite. Coconut shell reinforced composite was prepared by compacting epoxy resin matrix with 10% to 30% volume fraction of coconut shell fibres. They found that tensile strength of the composite increases with increase in coconut shell fibre content [9]. Haneef et al. (2013) developed hybrid bio polymer matrix composites. They used high density polyethylene as polymer matrix, and titanium Oxide (TiO_2) & Alumina (Al_2O_3) as reinforcing material. They found an appreciable improvement in the mechanical properties with tensile strength of 16.7 MPa, bending strength of 12 MPa and shore hardness of 55 (shore D). Further corrosion test showed that there was no corrosion observed on the specimens after 24 hours at pH value of 7 [10]. Jagadish et al. (2015) evaluated mechanical properties of carbon fiber polymer composite material with +/- 0° to 90^0 orientations used as implant material. Specimen is formulated according to ASTM standards. As a consequence these results compared with mechanical properties of femur bone [11]. Present study focuses on the synthesis of hybrid bio polymer matrix composites using low density polyethylene as matrix material with reinforcing material namely, alumina and titanium oxide.

2. Materials and Methods

From the literature review it was observed that there are three grades of commercially available polyethylene namely, low density, high density, and ultra high molecular weight polyethylene (UHMWPE). UHMWPE has less ductility and fracture toughness than those of other classes of polyethylene. But low density and high density polyethylene have better packing of linear chains and high cross-linking levels which results in their increased crystallinity and mechanical properties. Thus in the present study commercially available low density polyethylene (LDPE) was used as polymer matrix material. But these LDPE’s have less wear resistance and which can be improved by adding metallic or/and ceramic reinforcement material in the polymer matrix. Hence, in the present study alumina (Al_2O_3) and titanium oxide (TiO_2) were used as the reinforcing material. In the present study LDPE in granule form was used whereas Al_2O_3 and TiO_2 powder of 325 mesh size was used for the synthesis of polymer composites. It is necessary to have different composition of the same composite material to get the best result in mechanical properties with respect to strength of that same composite material. For this percentage by weight of each matrix material, Al_2O_3 & TiO_2 powder materials along with a surfactant (noninphinoethoxylate) material was used for the synthesis of composite material. The weight percentage of LDPE, Al_2O_3 and TiO_2 were decided based on the literature review, Haneef. M.et al., (2013). Table 1 shows the percentage of matrix and reinforcing material used for synthesis of polymer-metallic/ceramic composites. Standard test specimens were prepared as per the ASTM standard. Tensile test specimens were prepared as per ASTM D638 standards, flexural test specimens were prepared as per ASTM D790 standard and impact test specimens were prepared as per ASTM D256. In these Impact test specimen and Flexural test specimen having dimensions 64 mm x 13 mm x 3.5 mm and 127 mm x 13 mm x 3.5 mm respectively and tensile test specimen has dimensions 63 mm x 9.53 mm x 3.5 mm with cross section width 9.53 mm and radius of 12.7 mm as shown in figure 1.

Table 1. Composition of composite material.

Sr. No.	LDPE (%)	TiO_2 (%)	Al_2O_3 (%)
1	85	10	5
2	80	10	10
3	75	10	15
4	70	10	20

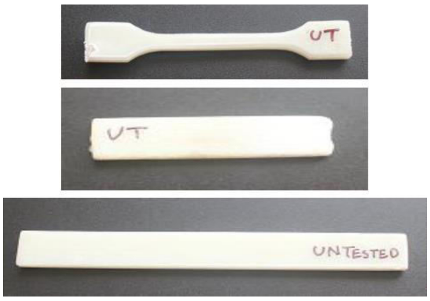

Figure 1. Test specimens as per ASTM standard

Vertical hand operated injection moulding machine was used for specimens preparation. In the working of the injection moulding, the raw material is injected through the hot barrel into the mould where it takes the inverse shape of it. It is possible to have the multiple cavity mould rather than the single cavity mould, which saves the time as well as the raw material. Figure 2 show the injection moulding machine along with multiple cavity mould.

Figure 2. Injection moulding machine along with mould

Tensile and flexural test were performed using VEEKAY TESTLAB, MUMBAI make with range of load varying from 0 to 10000 N with least count of 100 N. Impact test was performed using ADVANCE EQUIPMENTS, MUMBAI make with range 0 to 10 J with least count of 0.0001 J. Hardness of the synthesised polymer composites were measured using shore hardness D-scale as per the ASTM D2240 standard. DUCOM make pin-on-disc sliding wear testing machine was used to understand the dry sliding wear characteristics of the hybrid composite specimens. After each test coefficient of friction (COF) and height loss was recorded. As per ASTM standards the pin of ø8 x 32 mm length was used for tribotest. The dry sliding wear tests were conducted as per ASTM G99- 95 standards. The pin was cleaned with acetone and its initial mass was measured using a digital electronic balance. The pin is then held pressed against the rotating EN-32 steel disc (counter face) with a hardness of 65 HRC during the test. Tribological test were carried out with normal load of 50 N, track diameter of 75 mm, sliding speed of 350 rpm and the entire test were carried out for 15 mins duration. These test parameters were decided based on the literature review. Further, corrosion test was carried out on the composites as per salt spray test according to ASTM B117 standard.

3. Result and Discussions

This study depicts the mechanical behaviour of LDPE + Al_2O_3 + TiO_2 polymer composites. The specimens were synthesised using injection moulding machine and the test were carried out as per the ASTM standards. This section discusses the results of mechanical properties of developed composites in terms of tensile strength at ultimate, flexural strength at 7.8 mm deflection, impact strength at breaking point, shore hardness D-scale, wear/cof test and corrosion test.

3.1 Tensile strength

Tensile strength for the composites was calculated at ultimate point and is plotted in the bar chart as shown in figure 3. Here alumina (Al_2O_3) is varied from 5% to 20% in a step increase of 5% and titanium oxide (TiO_2) is kept constant at 10%. It can be seen from the figure that tensile strength increases with the increase in the percentage of alumina. This may be due to the presence of hard and stiff alumina particles in the composite material. Hence the load carrying capacity of the composite material increases. The maximum tensile strength obtained was 12.65 MPa for LDPE + 20% Al_2O_3+10% TiO_2.

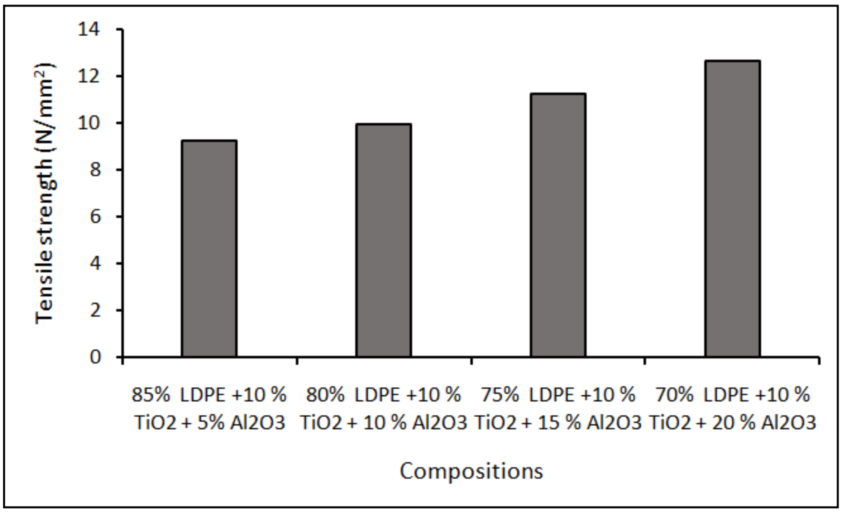

Figure 3. Tensile strength with varying percentage of reinforcing material

3.2 Flexural strength

Flexural strength for the composites was calculated when a deflection of 7.8 mm was achieved and is plotted in the bar chart as shown in figure 4. It can be seen from the figure that flexural strength increases with the increase in the percentage of alumina. This may be due to the presence of hard and stiff alumina particles in the composite material. Alumina particles resist the deformation of the composite material, thereby the flexural strength increases. The maximum flexural strength obtained was 15.09 MPa for LDPE + 20% Al_2O_3+10% TiO_2.

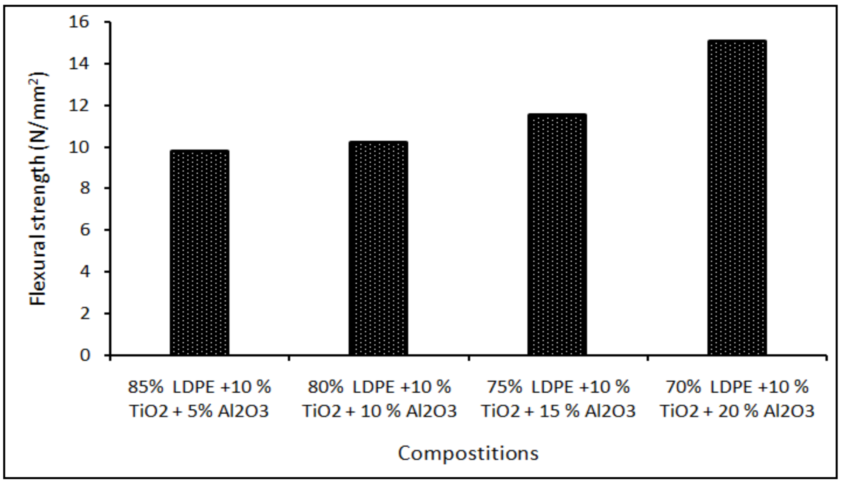

Figure 4. Flexural strength with varying percentage of reinforcing material

3.3 Impact strength

Impact strength of the composites was calculated at the breaking point and the energy observed before failure was noted for estimating the impact strength of the composites. Figure 5 shows the variation of impact strength with the change in percentage of alumina. It can be seen that the impact strength decreases as the alumina particles in the composites increases. As alumina is hard and stiffer material, it increases the tensile and flexural strength and decreases the ductility of the material. As ductility decreases the energy observed by the material before failure decreases, hence the impact strength decreases. The maximum impact strength obtained was 39.11kJ/m^2 for LDPE + 5% Al_2O_3+10% TiO_2.

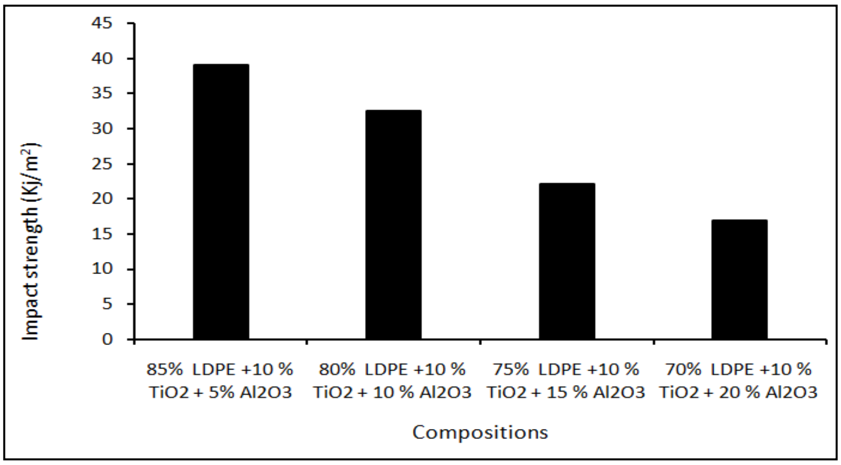

Figure 5. Impact strength with varying percentage of reinforcing material

3.4 Hardness

Figure 6 shows the shore hardness values of polymer composites with varying percentage of alumina. Hardness was measured at three different locations on the specimens and average values are used to draw the graphs. With the addition of alumina particles in the matrix, increases the hardness of the composites. This shows that the existence of alumina particles in the matrix of composites which improved the overall hardness of the material. It is due to the fact that the reinforcing particles being hard, contributes to increase in the hardness of the composites. The presence of harder and stiffer reinforcing material leads to increase the resistance to plastic deformation. The maximum hardness was obtained for LDPE + 20% Al_2O_3+10% TiO_2, having shore hardness value of 55.

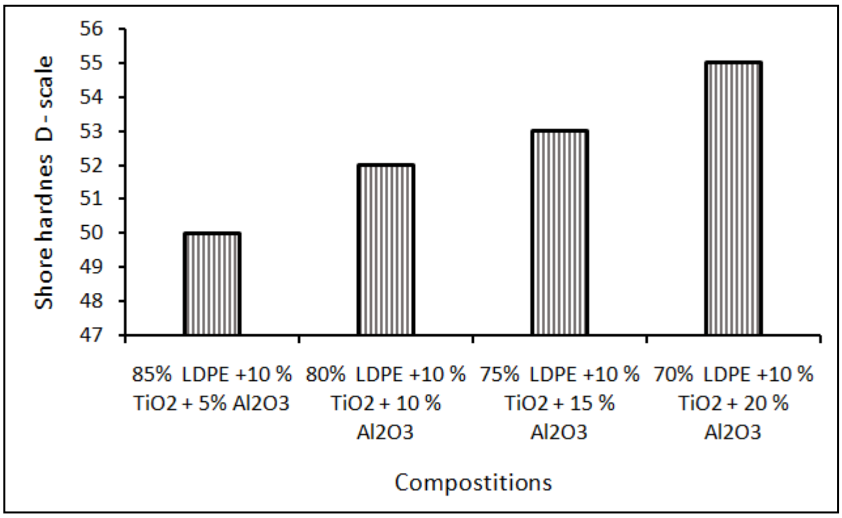

Figure 6. Hardness with varying percentage of reinforcing material

3.5 Tribological test

Tribological test consists of analysis of coefficient of friction and wear in terms of height loss. Triboloical test were carried out in dry condition using pin-on-disc tribometer. The test parameters considered for the study are: normal load of 50 N, track diameter of 75 mm, sliding speed of 350 rpm and the entire test were carried out for 15 mins duration. Figure 7 and 8 shows the coefficient of friction and wear of the composite materials.

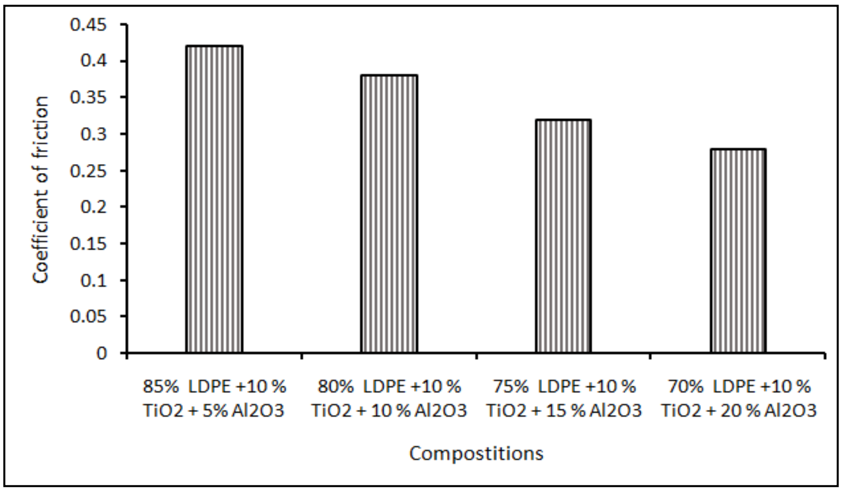

Figure 7. COF with varying percentage of reinforcing material

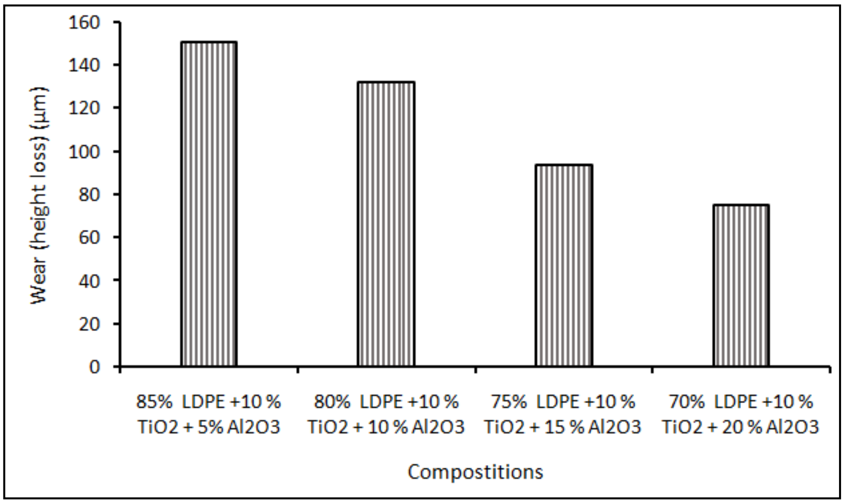

Figure 8. Wear with varying percentage of reinforcing material

It can be seen from figure 7 and 8 that the coefficient of friction and wear rate decreases with increase in the percentage of alumina particles in the composites. The minimum coefficient of friction (0.28) and wear (75 µm) was obtained at LDPE + 20% Al_2O_3+10% TiO_2. Wear of the composite material is less due the presence of alumina particles which increases the wear resistance of the composite material. The presence of harder and stiffer alumina particles leads to increase the resistance to peel off or scratch of the composite material. The minimum value for coefficient of friction may be due to presence of alumina particle which helps in rolling friction instead of sliding friction.

3.7 SEM image of fractured surface

The composite having the composition of LDPE + 20% Al_2O_3+10% TiO_2 was observed under the scanning electron microscopy for understanding the distribution of the reinforcing particles in the polymer matrix and to get the information about the type of fracture after the tensile test. Figure 9 shows the fractured surface observed at 200X magnification. It is clear from the SEM image that there is a homogeneous distribution of reinforcing particles in the matrix of the polymer and there are no casting defects observed. Also there is a proper bonding between the matrix and reinforcing particles. This enhances the mechanical properties of the composites materials. Further, the image shows that the composite fails by brittle fracture.

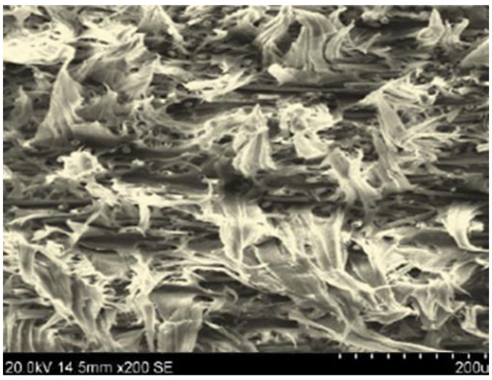

Figure 9. SEM image of fractured surface at 200X

3.8 Corrosion test

Corrosion test was carried for all the developed composites as per the salt spray test according to ASTM B117 standard. The test was carried out with 5% NaCl solution in distilled water with 1.40 ml volume of solution collected per hour per 80 cm^2 area maintained at 35º ± 2 ºC. After a time period of 48 hours and pH value of 7, no corrosion was observed for all the specimens.

4. Conclusions

In this study an attempt had made to synthesis a polymeric bio-composite for orthopaedic application such as implant material in the bone surgery. Following conclusions can be drawn based on the results of present study.

1. Tensile strength, flexural strength and hardness increases with increase in the percentage of alumina in the composite material. Maximum tensile strength (12.65 MPa), flexural strength (15. 09 MPa) and hardness (shore hardness number 55) was achieved when composite having composition of 70%LDPE+10%TiO_2 +20%Al_2O_3 was used.
2. SEM image shows a homogeneous distribution of reinforcing particle and proper bonding between matrix and reinforcement. Further, there were no casting defects observed.
3. Impact strength decreases with increase in the percentage of alumina in the composite material. Maximum impact strength (39.11 kJ/m^2) was obtained at 85%LDPE +10% TiO_2 +5% Al_2O_3.
4. Coefficient of friction and wear decreases with increase in the percentage of alumina in the composite material. Low coefficient of friction (0.28) and wear (75 µm) was observed at 70%LDPE +10% TiO_2 +20% Al_2O_3.
5. No corrosion was observed on the samples after time duration of 48 hours at a pH value of 7.

Injection moulding process is a successful fabrication technology for preparing samples made of biomaterial without any casting defects. Based on the results of various tests, it can be suggested that, 70%LDPE +10% TiO_2 +20% Al_2O_3 polymer matrix composite could be used as a suitable biomaterial in orthopaedic applications and can be applied on hard and soft tissues of implantable materials. Polymer matrix composite (70%LDPE +10% TiO_2 +20% Al_2O_3) is biocompatible, based on the results of wear and corrosion test.

5. References

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