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Research article

Development of High-Density Polyethylene-Hydroxyapatite-Carbon Fiber (HDPE-HA-CF) Polymer Composites for Human Bone Replacement

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A B S T R A C T

Demand for bone substitutes is rising due to factors like aging populations, injuries, and diseases. Polymer composites offer a promising alternative to traditional grafts, with hydroxyapatite and carbon fibers enhancing mechanical properties in this research. High-density polyethylene-hydroxyapatite-carbon fiber (HDPE-HA-CF) composites are made with extrusion and semi-injection molding processes. The morphological characteristics of the cross-section area and mechanical test are performed. Experimentation examined the effect of hydroxyapatite and carbon fibers as reinforcing materials in various weight percentages (5%, 10%, 20%, 30%, 40%) on the HDPE-HA-CF composite properties: increased tensile strength, modulus, flexural properties, and hardness; decreased elongation and impact strength with higher reinforcement weight percentage. Depending on the load transfer and uniform distribution of the particles, 40 weight percentage HA-CF reinforced composites revealed the highest strength, modulus, and hardness, but considerably lower elongation and impact strength of mechanical behaviors for the HDPE matrix among the other compositions. The research findings would provide important lessons for developing durable bone replacements using advanced polymer composites.

1. INTRODUCTION

The compact (cortical) bone and the cancellous (trabecular/spongy) bone are the two different forms of bone that make up the key structural elements of the human body. In contrast to the cancellous bone, which is less dense and extremely porous and provides only 20% of the adult connective tissue, the compact bone is tightly packed, hard, and dense [1]. When damaged, they have a special ability to regenerate. When injury exceeds a threshold magnitude, however, bone cannot regenerate through normal physiological mechanisms [2].

Due to decreased physical activity, genetic flaws, age, aging, sickness, trauma, degeneration, accidents, and rising obesity, the prevalence of bone problems has dramatically increased around the world. Osteoporosis causes millions of cases of deteriorating bone, and individuals who have low bone mass are more susceptible to illnesses. Human bone replacement is a critical field in orthopedic surgery, aiming to restore the function and structure of damaged or diseased bones. Traditional bone replacement materials, such as metals and ceramics, have been used for decades [3-5], but they have certain drawbacks, including poor biocompatibility and stress shielding. As a result, research has shifted towards developing advanced biomaterials that can better mimic the mechanical and biological properties of natural bone. Research is still being done to find materials that can withstand the mechanical and biological demands of permanent surgery and high wear resistance. In other words, the need for artificial biomaterials with strong biocompatibility to bone is growing due to the aging of the population, a variety of skeletal issues caused by accidents and diseases, and the rapid advancement of health and medical treatment technologies [6-8].

The field of orthopedic surgery has witnessed remarkable progress in the development of materials for human bone replacement. However, the ideal material for human bone replacement remains an ongoing challenge due to the complex requirements of mechanical properties, biocompatibility, and biodegradability. In recent years, polymer composites have emerged as promising candidates for bone replacement due to their versatility and potential to mimic natural bone properties. For bone restoration, a substance with high bioactivity and robust biocompatibility would be appropriate. They must also possess mechanical properties that are comparable to those of natural bone. The aim of this research was to create a new composite material that may be used to repair bone defects.

For decades, hydroxyapatite (HA; Ca10(PO4)6(OH)2) has been a popular option for bone replacement material [9-11]. Synthetic HA has demonstrated remarkable biocompatibility in vitro and in vivo due to its resemblance to the mineral component of real bone [12]. Nevertheless, because of the hydroxyapatite's weak mechanical properties, such as low tensile strength and brittleness, the clinical therapies have only proven effective for long-bone fractures or severe load-bearing [13, 14]. One way to improve the biomechanical characteristics of pure HA is to include a reinforcing phase such as carbon fibers (CF). As they have such great qualities as being lightweight and having a high strength and modulus, carbon fibers (CF) have been frequently employed as reinforcement for ceramic and polymeric matrices [15, 16]. Furthermore, a number of research studies [17, 18] suggest that carbon fibers are biocompatible both in vitro and in vivo, and they have already been employed as biomaterials [19-21]. Because of its high strength, great biocompatibility, and toughening capabilities, carbon fiber (CF) is regarded as the ideal reinforcing material [22, 23]. In the other, due to the light weight, reduced inflammatory response, simplicity of production, great biocompatibility, and reasonably cheap cost, polymers have proven useful in bone-related applications. Some polymers, such as HDPE, can function as the natural collagen in bone. High-density polyethylene (HDPE) shows excellent mechanical properties, and it has been widely used as an implant material for bone reconstruction [24]. So, HDPE could be a better choice as a matrix phase material.

To overcome the drawbacks of conventional bone replacement materials, the present research focuses on the development and investigation of innovative polymer composites, namely high-density polyethylene-hydroxyapatite-carbon fibers (HDPE-HA-CF), and that material could be used commercially for the betterment of humankind.

2. MATERIALS AND METHODS

2.1 Materials

The thermoplastic polymer HDPE (Chevron Philips), used as a matrix, was collected by Al Jubail Polymers Company, Kingdom of Saudi Arabia, in the form of homopolymer pellets. The HDPE had a specific gravity of 0.88–0.968 g/cm^2, melting temperatures ranging from 120 to 180°C, and a crystallinity of 75%. Hydroxyapatite (HA) was collected from Shandong Honrel Co., Limited, Zibo City, Shandong Province, China. The inorganic mineral hydroxyapatite (HA) is found in human teeth and bones [25]. It is a calcium

apatite mineral that occurs naturally and makes up over 50% of bone weight, which explains its superior osteoconductive and osteointegrative qualities [26-28]. Particularly, it has a hexagonal structure [29, 30] and a stoichiometric Ca/P ratio of 1.67, which is identical to bone apatite [31, 32]. Its molecular formula is $Ca_5(PO_4)_3(OH)$, its molecular weight (g/mol) is 502.31, and its particle size (D50) is 200 nm with pH=6.5-8.5. Carbon fibers were collected from Shenzhen Yataida High-Tech Co. Ltd., China. Its powder diameter is 7.0 μm, tensile strength is 3.5 GPa, Young's modulus is 230 GPa, average fiber length is 75-80 μm, mesh is 200, and carbon content is >99%.

2.2 Composites Preparation Procedure

For preparing a composite, a definite amount of matrix and reinforcement is needed. In this research, five types of composites were prepared, and the ratios of HDPE, HA, and CF are provided in Table 1.

Table 1. Composition of HDPE-HA-CF polymer composite.

Sample	Matrix (HDPE) (wt.%)	Reinforcement (wt.%)		
		HA	CF	Total
1	95	4.38	0.62	5
2	90	8.75	1.25	10
3	80	17.5	2.5	20
4	70	26.25	3.75	30
5	60	35	5	40

To prepare one type of composite each time, a 200 gm mixture of matrix and reinforcement was used. For 5 wt.% reinforcement, the taken weight of the matrix was 190 gm and the reinforcement was 10 gm. Again, for 10 wt.%, 20 wt.%, 30 wt.%, and 40 wt.%, the taken weight of the matrix was 180 gm, 160 gm, 140 gm, and 120 gm, respectively, and the reinforcement was 20 gm, 40 gm, 60 gm, and 80 gm, respectively. Here reinforcements are taken at the desired amount and mixed properly. Then this reinforcement mixture is mixed with matrix (HDPE). Extrusion means squeezing out by applying pressure. In the experiment it is used to carry out the composite materials through small openings. An electrically operated customized extruder is used for this purpose. It consists of a hopper, nozzle, motor and gear controller, rotating shaft, heating coil, and temperature display board. Coil temperature is set at the required point depending on the type of composition of composites. There are three display units for showing set value and present value, named SV and PV, respectively. When the PV reaches SV, then the heating coil is automatically stopped, and then, with the help of the motor, composite materials are squeezed out through the nozzle.

At first high-density polyethylene (HDPE), hydroxyapatite, and carbon fibers were mixed properly and gradually poured into the hopper in modest amounts each time. These then came into contact with the rotating shaft, where they mixed with each other and were heated by the coils that were present over the shaft. The set temperatures of the 1st, 2nd, and 3rd heating coils were 150°C, 200°C, and 250°C, respectively. The speed of the shaft was maintained by the motor, which helps to squeeze out the mixture through the nozzle. Shaft speed is carefully controlled for proper and uniform mixing. Then the molten composite materials are dried out. Then these materials are cut into small pieces and poured into the semi-injection molding machine for further mixing and to get the desired size of polymer composites for characterization [33].

2.3 Characterization

Microstructural analysis

The interfacial bonding between reinforcement and the HDPE matrix in manufactured composites was examined using an Optical Microscopy (OM) (ML-803, Taiwan) and Scanning Electron Microscope (SEM) (JSM-7600 F) supplied by JEOL Company Limited, Japan. Composite samples were gold coated before morphological analysis under SEM.

Mechanical Test

HDPE-HA-CF-based polymer composites were tested for mechanical characterization (tensile, flexural, impact, and hardness). All tests should be carried out by using ASTM standards. The universal testing machine (UTM) (H5OKS, Hounsfield, USA) is suitable for many mechanical tests of polymer composites. Tensile properties were usually measured at a speed of 100 mm min^{-1}. The sample is elongated through the moving crosshead. The load cell shows the magnitude of the applied load

on the sample, whereas the extensometer measures the elongation of the sample. During the tensile testing, deformation occurs at the central region of the sample, which contains a uniform cross-sectional area along its length [34]. A tensile test was performed according to ASTM for determining tensile strength using a universal testing machine (UTM) Model D638-01. The result of tensile strength obtained by testing three specimens and the mean value was taken as data for each sample. The specimen dimension was length 63.5 mm, width 9.53 mm, and thickness 4 mm, respectively. A flexural test was performed according to ASTM D790-00 to determine flexural strength using a three-point bending mode testing machine at the constant deflection rate of 10 mm/min. The result of flexural strength obtained by testing three specimens, and the mean value was taken as data for each sample. The specimen dimension was length 80 mm, width 12.5 mm, and thickness 4 mm, respectively. Here, dynamic Charpy impact tests were performed on notched composite specimens. The samples were prepared from the manufactured composite and milled to the standard size. The test specimen was supported as a vertical cantilever beam and broken by a single swing of a pendulum. The pendulum strikes the face of the sample. The impact test was performed by using the impact tester MT 3016 according to ASTM D 6110-97 for determining the impact strength of the materials. The result of impact strength obtained by testing three specimens and the mean value was taken as data for each sample. The specimen dimension was length 70 mm, width 10 mm, and thickness 4 mm, respectively. The hardness of the composite was measured using a Shore hardness tester (Durometer, AD-100-A, USA) in the D scale. Here, in the graph, the error bar represents the ± standard error based on three specimens.

3. RESULT AND DISCUSSION

In this research, several weight percentages of reinforcement—5%, 10%, 20%, 30%, and 40%—were used with a different matrix. Reinforcement was provided by Carbon Fibers (CF) and Hydroxyapatite (HA) within matrixes of High-Density Polyethylene (HDPE). They were carefully combined and weighed. Five different reinforcement compositions were used to achieve the progressive features of the composites; in each case, a well-mixed, uniform dispersion of particles was maintained.

3.1 HDPE-HA-CF Polymer Composites

Morphological Observation of the Composites by Optical Microscope (OM)

An optical microscope (OM) can be used to study the morphological characteristics of any manufactured composite sample. In this investigation, the produced composite samples were examined carefully using an optical microscope at a 40x magnification to determine the particle distribution, agglomeration, surface morphology, and presence or absence of clusters, cracks, and voids.

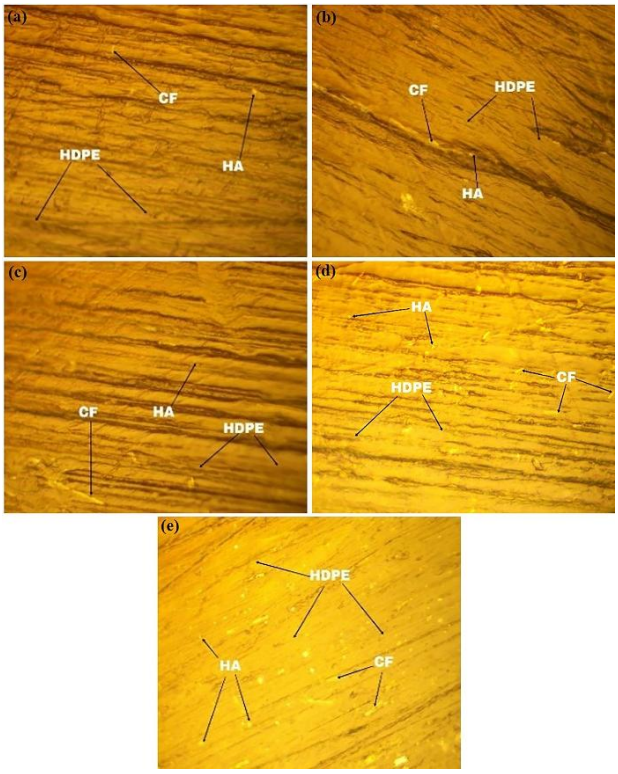

Fig. 1. Optical micrographs of composite cross-section: (a) 5 wt.% reinforcement composite, (b) 10 wt.% reinforcement composite, (c) 20 wt.% reinforcement composite, (d) 30 wt.% reinforcement composite, and (e) 40 wt.% reinforcement composite of HDPE-HA-CF polymer composites.

Optical microstructural observations of the HDPE-HA-CF polymer composites from figure 1 (a, b, c, d, and e) were first conducted to confirm the distribution of the carbon fibers and hydroxyapatite particles within the polymer matrix. Additionally, the integrity of the interface frontier, particle agglomeration, and the presence or absence of voids were also examined. The composites' optical micrographs revealed that there are no significant gaps or cracks and

that the hydroxyapatite and carbon fiber particles are evenly distributed throughout the matrix phase. Large voids and reaction products are absent, and the interfaces are excellent and crisp with intact integrities [35].

Morphological Observation of the Composites by Scanning Electron Microscope (SEM)

Observation of the cross-section of the composites by SEM can give rise to information about the sample, including the external morphology (texture), interfacial adhesion, crystalline structure, and orientation of the materials making up the sample. Figures 2 (a, b, c, d, and e) illustrate the morphology of the 40 wt.% HDPE-HA-CF polymer composites.

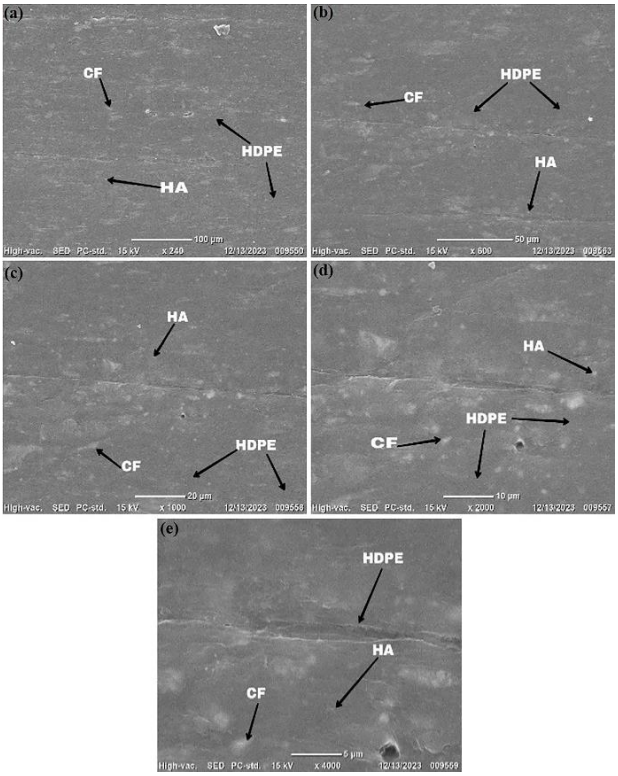

Fig. 2. Scanning electron micrograph shows the distribution of HA and CF particles in 40 wt.% reinforcement of HDPE-HA-CF polymer composites (a) 100 µm scale, 240x magnification, (b) 50 µm scale, 600x magnification, (c) 20 µm scale, 1000x magnification, (d) 10 µm scale, 2000x magnification, and (e) 5 µm scale, 4000x magnification.

Figure 2 (a, b) shows that the carbon fibers and hydroxyapatite were evenly distributed throughout the matrix phase, with no visible gaps in the interfacial area between them. Additionally, (c) illustrates that the surfaces of the fibers appear to be coated in HDPE. This demonstrated a well-bonded contact. An enhancement in mechanical qualities further supported the strong adhesion at the particle-polymer matrix interface, which is associated with the encapsulation of polymer with hydroxyapatite and carbon fibers. In the matrix, carbon fibers and hydroxyapatite were layered on top of one another. It was observed that the percentages and polymers of HA and CF reinforcement were extremely well mixed. A detailed examination of the sample from figure 2 (d, e) indicates that there were no pores, cavities, or voids. It appears that composites with a well-bonded matrix and reinforcement are moving toward substantially intrinsic qualities and multifunctional textures. The findings imply that the intermediate load-bearing components of a composite are the matrix's primary functioning elements, and the reinforcement's role is to contain the matrix and so boost the composites' strength. Efficient load distribution and transfer from the matrix to the reinforcement are made possible by a well-bonded interface [36].

3.1 Mechanical Test

3.1.1 Tensile Strength

In figure 3, it is demonstrated that the polymer composite's tensile strength rises somewhat with the reinforcement. Improved tensile strength is the result of smaller particle size, homogeneous distribution, and appropriate reinforcement-matrix interaction. By physically interacting with the matrix, rigid particles impose mechanical constraints on the mobility or deformability of the matrix. This prevents stress concentration from forming around the reinforcements in stressed composites and enables the composite to withstand higher applied loads [37, 38].

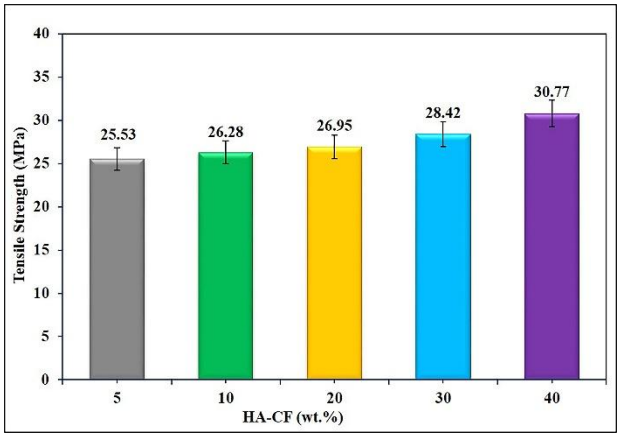

Fig. 3. Tensile strength vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

A 40 wt.% loading of reinforcement content resulted in the maximum tensile strength of 30.77 MPa. Therefore, under tensile loading, it is expected that the composite's particles will provide a greater load-carrying capability.

3.1.2 Young's Modulus

Figure 4 displays the Young's modulus value increased with the increase of the reinforcement particles. At 40 wt.%, it reaches a maximum of 1876 MPa. Young's modulus is influenced by a number of variables, including elongation, temperature, impurity effect, and stress strain.

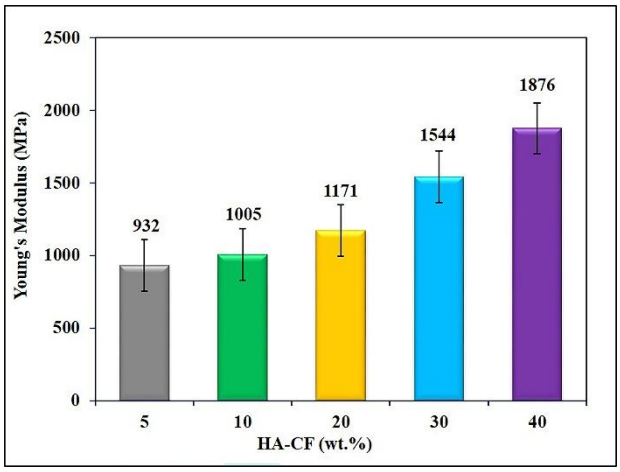

Fig. 4. Young's modulus vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

It has been noted that when reinforcement loading increased, so did Young's modulus. The inclusion of stiff particles like carbon fibers and hydroxyapatite raises the elastic modulus [39, 40]. According to other investigative evidence and conventional equations of micromechanics of composite materials, the following rigid particles are likely to make the material extremely stiff [41, 42].

3.1.3 Elongation at Break (%)

Figure 5 shows that when the loading of the reinforcement increased, the elongation characteristics decreased significantly. At 5 wt.% reinforcement load, it is 13.7%, and at 40 wt.% reinforcement load, it is 3.63%, which is about a 73.5% decrease. For every particle, it was found that the values of elongation at break fell as loading increased. The rationale for this behavior is that when strain is applied, the adherence of particles to the polymer matrix stiffens the polymer chain, which reduces elongation and causes the material to stretch [43].

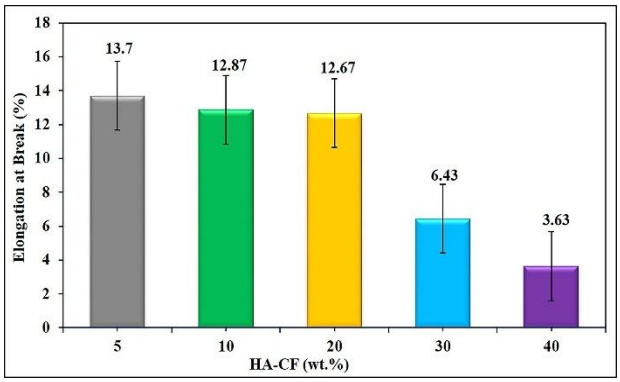

Fig. 5. Elongation at break vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

Conversely, elongation has an inverse relationship with Young's modulus. Consequently, the elongation of the manufactured composites reduced as the proportion of reinforcement increased [44-46]. The manufactured composites had a lower percentage of elongation at break because, all things considered, they were stiffer than the virgin HDPE matrix.

3.1.4 Flexural Properties

A three-point bending mode testing apparatus was used in a flexural test in accordance with ASTM D 790-00 to measure flexural strength at a steady deflection rate of 10 mm/min. Three specimens were tested for flexural strength, and the mean value was taken as the data for each sample.

3.1.4.1 Flexural Strength

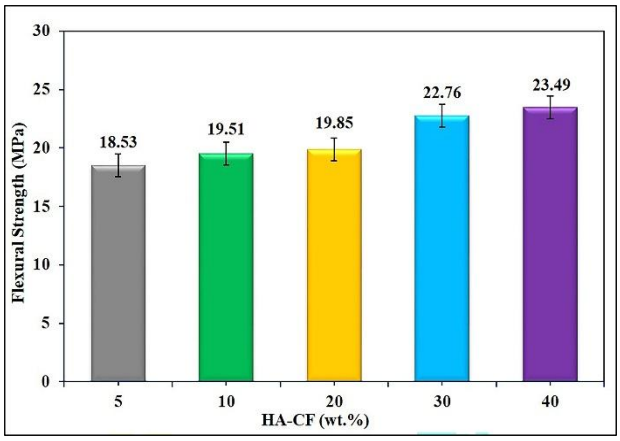

Fig. 6. Flexural strength vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

The impact of the reinforcing content on the HDPE-HA-CF polymer composites' flexural strength is depicted in figure 6. This discovery makes it abundantly evident that the insertion of HA and CF particles into the polymeric matrix develops the flexural strength. Flexural strengths of HA-CF-reinforced 5% and 40% polymer composites are 18.53 MPa and 23.49 MPa, respectively. Here the range of flexural strength is with an increment of 26.76%. The explanation is improved processing and mixing methods, which even have an impact on the distribution of particles in the composites [47– 49]. A further explanation could be that the stiff particles physically interact with the matrix to put mechanical limitations on the matrix's deformability or mobility.

3.1.4.2 Flexural Modulus

Figure 7 displays the HDPE-HA-CF polymer composites' flexural modulus values at various reinforcement loadings. This figure illustrates how the reinforcement loading grew along with the flexural modulus.

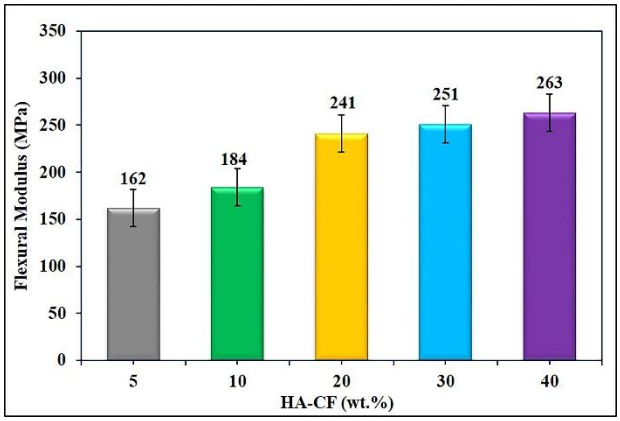

Fig. 7. Flexural modulus vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

The flexural modulus is 162 MPa at 5 wt.% and 263 MPa at 40 wt.%. The polymer composites have seen an increase in flexural modulus of roughly 62.34%. A higher modulus is obtained when reinforcement (HA, CF) is added to the virgin HDPE matrix [50].

3.1.4 Impact Properties

Impact strength can be defined as a material's capacity to absorb energy in the event of a rapid load applied to it, as well as the point at which the material becomes brittle instead of ductile [51].

3.1.5.1 Impact Strength

The impact strength of the polymer composites at various HA and CF reinforcing percentages is displayed in figure 8. The impact strength of 5 wt.% HDPE-HA-CF polymer composites is 17.85 Kj/m^2, whereas the impact strength of polymer composites, which make up 40 wt.%, is 5.05 Kj/m^2. In comparison to 5 wt.% to 40 wt.% HDPE polymer composites, it demonstrates a reduction in impact strength of up to 71.71%.

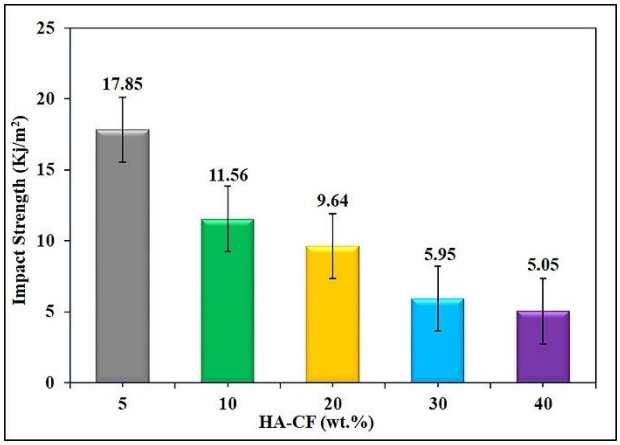

Fig. 8. Impact strength vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites

Because agglomeration and inter-particle spacing govern impact strength upgrading, the best results are achieved at modest concentrations of stiff reinforcement. Perfect adherence of stiff particles causes more matrix constraint due to matrix contact with the reinforcing surface, further embrittling the material [49, 51]. This could be the cause of the polymer composite's declining impact strength as the content of reinforcing increases.

3.1.6 Hardness Properties

A material's hardness determines how resilient it is against dents, abrasions, and stresses. This study's objective was to determine the material's relative hardness using a Shore D Hardness Tester.

3.1.6.1 Hardness

In figure 9, the HDPE-HA-CF polymer composites' hardness behavior is displayed. When reinforcements are evenly distributed throughout the polymeric matrix, the resulting polymer composite's hardness is balanced. The

fraction of HA and CF particles appears to be rising in tandem with an apparent increase in hardness. In room-temperature reinforced polymer composites, the hardness rose from 70 shores at 5 wt.% to 79 shores at 40 wt.%, which is about a 12.86% increase. Particulates that are typically stiff, such as HA and CF, may be the cause of this [47]. By strengthening the physical relationship between the reinforcement and matrix, the reinforcement particles have reinforced the pores in the polymer matrix, strengthening the composites and reducing plastic flow. The composite's hardness will increase as well because of the increased interaction between the particles and matrix [52].

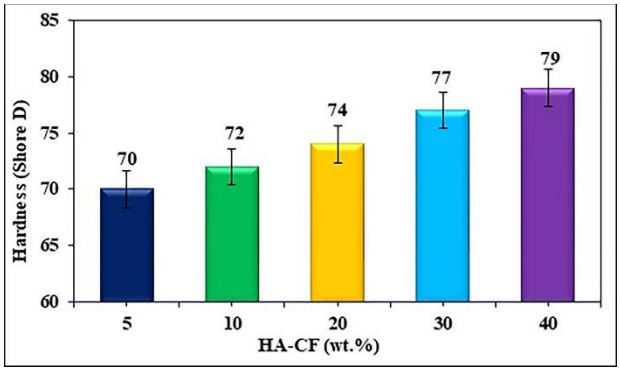

Fig. 9. Hardness vs. reinforcement (wt.%) curve for HDPE-HA-CF polymer composites.

Overall, as the proportion of reinforcement increases, so do the tensile strength, tensile modulus, flexural strength, flexural modulus, and hardness values of HDPE-HA-CF polymer composites. Conversely, when reinforcing percentages grow, HDPE-HA-CF polymer composites' impact strength and elongation at break values fall. It's clear from the debate above that this polymer composite is better.

4. CONCLUSION

Nowadays, because of their superior qualities—such as being lightweight, having a high tensile strength, and being inexpensive—composite materials are taking the place of conventional materials. Composite materials have become increasingly important in response to the rapidly growing need for high-performance, lightweight materials. Because of their excellent specific strength and modulus, particle-reinforced polymer composites have found widespread use in engineering applications. Interest in lighter but durable composites to replace heavier metal parts has prompted a lot of research on particle-based composites. The mechanical characteristics of polymer composites reinforced with varying weight percentages of hydroxyapatite and carbon fiber powder are assessed in this work through processing and experimental tests. Here, 5 wt.%, 10 wt.%, 20 wt.%, 30 wt.%, and 40 wt.% hydroxyapatite and carbon fiber powder were used as reinforcement, and high-density polyethylene (HDPE) was used as a matrix. Using varying weight percentages of reinforcement, a novel form of HDPE-HA-CF composite was successfully fabricated using a customized extruder and adapted semi-injection molding machinery. Composites made with a semi-injection molding machine and customized extruder are more cost-effective than composites made with any other reputable machine or by other means. The HDPE-HA-CF composite's optical micrographs showed that there were reaction products and no significant voids. The scanning electron microscope (SEM) was utilized to prove the uniformity and particle distribution of prepared composites. SEM micrographs of HDPE-HA-CF composites showed that there were no voids, cavities, or larger pores, but some agglomerations of reinforcement particles were observed. It has been noted that the weight percentages of hydroxyapatite and carbon fiber powder have a significant impact on the mechanical performance of the HDPE-HA-CF composites, including their tensile and flexural qualities, hardness, impact strength, and elongation percentages. It was demonstrated by the experimental results that the mechanical characteristics of the HDPE-HA-CF composites generated at 40 weight percent of hydroxyapatite and carbon fiber powder loading are better to other weight percentages of powder loading for all fabricated composites. In conclusion, these synthesized polymer composites may find application in the development of bone substitutes after more biological investigation.

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