Mechancial Behaviour of Silver and Copper Nanoparticles When Incorporated With Poly-Lactic Acid Nanoparticles

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Cite this article:
Bobba, S., Malyadri, T., Leman, Z. et al. Mechancial Behaviour of Silver and Copper Nanoparticles When Incorporated With Poly-Lactic Acid Nanoparticles. J. Inst. Eng. India Ser. D (2024). https://doi.org/10.1007/s40033-024-00767-9

ABSTRACT

This current research work was performed on the integration of antimicrobial metal nanoparticles with thermoplastics and then to 3D print the obtained material. Mechanical properties such as tensile, compression and flexural were analyzed on the obtained specimens. The test specimens were developed of printed using fused deposition modelling method which involves material extrusion and the specimen parameters were taken based on the ASTM standards, Both the silver nanoparticles (AgNPs) and copper nanoparticles (CuNPs) were of 100 nm in size, 2% vol of AgNPs and CuNPs were added to Polylactic acid (PLA) granules, the nanoparticles were studied for concentration and scanning electron microscope (SEM) was utilized to investigate the composition of nanoparticles in Polylactic acid (PLA) granules.

Keywords: nanoparticles; thermoplastics; Mechanical properties; extrusion; ASTM standards

1. INTRODUCTION

Additive manufacturing contains a broad spectrum of techniques to turn the 3d designs into definite parts, with the help of its layer-by-layer process it builds a 3d model based on the data available. This could be similar to a printer printing images onto a paper. Additive manufacturing technology used design model from software such as CAD, this usually gives an advantage of modelling complex parts and shapes that cannot be processed by traditional machining processes, compared to subtractive manufacturing methods such as turning, drilling and other machining operations, additive machining uses no fixtures, requires no tool holders or molds or tooling. The wastage of material is more in traditional machining operations. Another significant advantage is near-net shaping feature, this reduces both the wastage and material usage. With the rise in need of sustainable development, additive manufacturing can play an important role in production with low raw material consumption, up to 75% and overall reduce in the carbon footprint.

T. N. A. T. Rahim et al. [1] have reviewed various polymers and the recent developments and progression in the FDM process in an venture to enhance the end production of 3D parts, alongside with present day challenges and the future direction. Zhai, Lados and Lagoy et al [2] have reviewed the additive manufacturing and the techniques involved in the manufacturing process, applications and materials used for specific technique.. Investigations and research regarding the static, dynamic and high-temperature properties of additive manufacturing materials should be studied more as per the suggestion made by the authors. Saleh Alghamdi, S, John, S, Roy Choudhury, N, Dutta N.K et al [3] have worked on various polymer materials used in additive manufacturing. Mechanical properties such as tensile modulus, tensile strength, and density for commercial polymers were obtained. Fiber composites have high strength, performance and durability which makes them preferable for various industrial application like production of automobile parts or musical instruments or sports equipment. Research work was performed by V. Shanmugam et al [4] on the fabrication of polymer-fiber composite and testing on the produced specimen followed by analyzing its performance. Fused deposition modelling was the chosen technique for the fabrication of polymers. Many ASTM standards were provided such that based on the material being polymer or composite, the

ASTM standard for testing can be chosen for tests like tensile, flexural, compression and impact. The article summarized mechanical performance of fiber reinforced composites over the years using FDM. It was observed that increase in fiber content and length, the strength also increased. it was observed that majority of researchers included and reported on tensile properties but for inclusion into modern applications more research regarding the flexural properties, impact strength and compression strength are also needed. Andrew Kvalsvig, Xiaowen Yuan, Johan Potgieter, Peng Cao et al [5] have used fiber reinforced thermoplastic components or specimens to print the 3D Specimen. for tensile specimens, ASTM D638 (standard method for tensile test of plastics). Nylon was used as the matrix material and fiberglass, HSHT fiberglass, carbon fiber, Kevlar fibers were used as fiber reinforcement that are embedded within thermoplastic polymer. The materials are available in filament form available in spools. for experimental analysis Taguchi technique was followed to determine the number of specimens, it was observed that the specimen with more fiber reinforcement had better tensile performance that specimens with less fiber reinforcement and also that the most influential variable was number of fiber layers. K.G. Jaya Christiyan et al. [6] has performed work on the ABS+hydrous magnesium silicate composite specimens for testing were printed using fused deposition modelling technique. for all the experiment a nozzle of diameter 0.6 mm was used, nozzle temperature was at 190°C during the extrusion process and the build platform was at 70°C and the infill density was 60%. The samples were prepared based on variations of layer thickness, printing speed and nozzle diameter. According to ASTM D638 tensile test was conducted and according to ASTM D790 flexural test was conducted. For each mechanical tests three samples were taken based on the variables. Both the mechanical tests were conducted on a universal testing machine with fixtures for both on a single test machine, it was observed that tensile and flexural strength were decreasing with respect to an increase in layer thickness, maximum values were reported for low layer thickness and printing speed as it gives better bonding between one layer and another reason for reduction in strength and stress variation could be due to delamination between the layers. Juan Domínguez-Robles et al. [7] has performed a collective research combined and proposed a method to combine LIG with poly(lactic acid) (PLA) for fused filament fabrication applications (FFF). PLA pellets were successfully coated with LIG powder and a biocompatible oil (castor oil). The resulting pellets were placed into an extruder at 200 °C. The resulting PLA filaments contained LIG loadings ranging from 0% to 3% (weight-weight percentage). The obtained filaments were successfully used for FFF applications.Humberto plaza et al. [8] have investigated antimicrobial properties in general and how metal nanocomposites are act against those microbes. The mechanisms of antimicrobial operation of nanoparticles are discussed by the researcher. Silver and copper metal nanoparticles, how they affect the bacteria under different circumstances are reviewed in the research conducted. Qinghao he et al. [9] have completed a survey that depicts about the printing of persistent carbon fiber composites utilizing melded affidavit displaying (FDM) strategy has resolved the issue of low mechanical execution of crude or short-fiber built up polymer parts manufactured by a similar cycle, because of the amazing explicit strength and firmness of nonstop filaments. The upsides of longitudinal malleable and flexural properties are unmistakably lower than those assessed by the standards of mixtures, while the Mode I interlaminar crack durability is likewise much lower than that of CF/PA6 overlays produced using the customary strategies.

Till current date, no details were reported on the production of the PLA film incorporated with silver and copper nanoparticles are available in the literature. So in the current research the specimens of silver nanoparticle infused PLA and copper nanoparticle infused PLA are designed using CATIA V5 software. The 3D models of the specimens are drawn. Prior to 3D printing step, the material chosen is drawn into filaments so that the spool can be fixed behind the 3d printer machine from where the extrusion head takes material to melt and layer on the build platform. Drawing file is converted to .STL file and the slicing of the file takes place. Cura was used as slicing software. Slicing basically gives the layering thickness, smaller the lower the layer thickness and finer the product. The filament drawn is sent through extrusion head with heaters and the material is layered according to the drawing file. The testing of Mechanical properties, Tensile, Flexural and Compression were done according to the ASTM standards. All the tests were done on one Ultimate Testing Machine (FIE UTES 40KN) that had setups for all tests. The data points for all the mechanical tests were collected and the respective load-displacement, stress-strain graphs were plotted to analyze and compare the mechanical properties of both metal nanoparticles in PLA.

2. MATERIALS AND FABRICATION

Any product's end usage in any industry usage involves some usage of mechanical loading hence, it is important to study the mechanical properties of the materials such that they can be used appropriately. Mechanical characteristics such as tensile strength, modulus, elongation, and impact strength are frequently used to select materials for a spectrum of uses. The mechanical tests conducted for both the silver-nano PLA and copper-nano PLA are tensile, compression and flexural. The tests were conducted according to the ASTM standards (ASTM D638, ASTM D695, ASTM D790 for tensile, compression and flexural respectively.) All of the test specimens were made from 3D CAD models as shown in Figure 1, and they were all made to the exact proportions stipulated in the applicable standard.

Figure 1: FDM machine (IEMAC MAGIC-HT-M)

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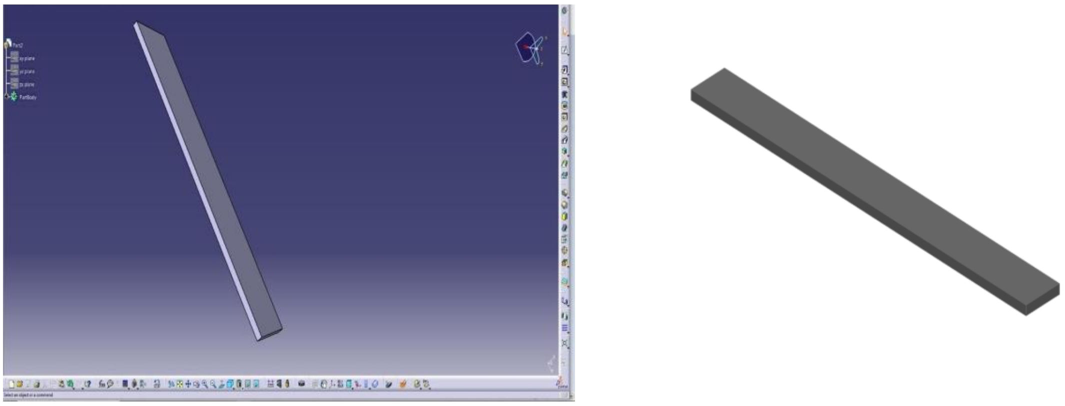
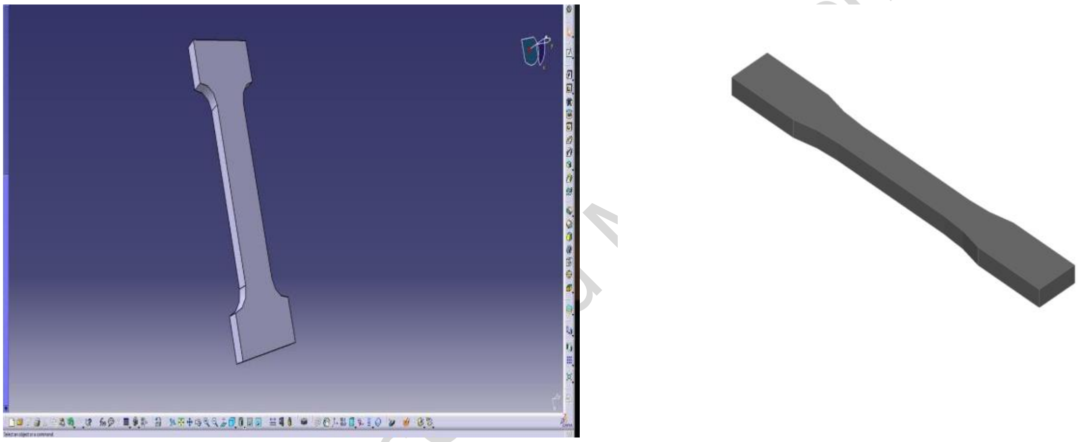
2.1 Fabrication of FDM parts:

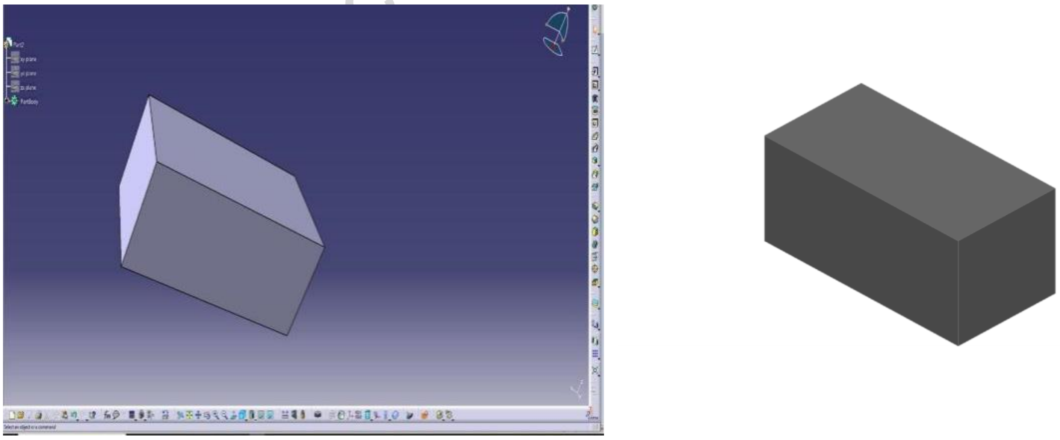
The wall thickness of the CAD model was offset during the pre-processing step to account for the metal foil thickness that would be noticed at the deposition phase. The models of standard specimens fabricated are shown in the below Figure 2.



(a) (b)



(a) (b)



(a) (b)

Figure 2: (a), (c), (e) are drawing models and (b), (d), (f) are .STL files of specimens

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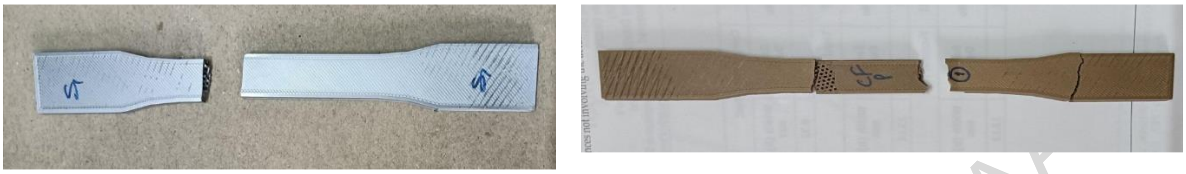
2.2 3D Printed Specimen Samples

Silver nanoparticles induced PLA and copper nanoparticles induced PLA specimens were printed by 3D printer “IEMAC MAGIC-HT-M" according to the ASTM standards as shown in the Figure 3,4,5 below. 3D printers can create complicated designs that fit through narrow areas or bend around structures, saving money on material. Unlike traditional manufacturing methods, these do not need heavy machinery or tooling and expensive tools. Instead of multiple sections, part can be printed as one eliminating labor and assembly cost.

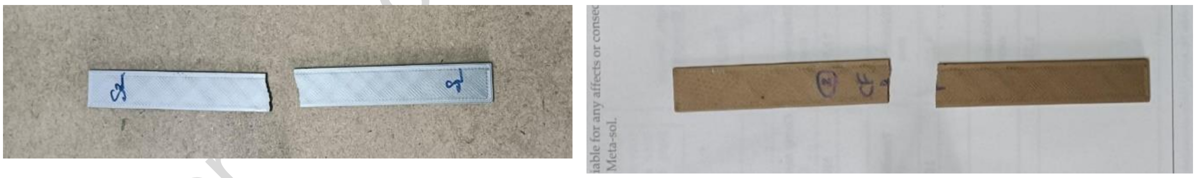


(a) (b)

Figure 3: Tensile specimens (ASTM D638) (a) silver nanoparticle induced PLA (b) copper nanoparticle induced PLA

(a) (b)

Figure 4: compression test specimens (ASTM D695) (a) silver nanoparticles induced PLA (b) copper nanoparticles induced PLA



(a) (b)

Figure 5: flexural test specimens (ASTM D790) (a) silver nanoparticles induced PLA (b) copper nanoparticles induced PLA

3. EXPERIMENTAL SETUP AND PROCEDURE

Work is done on silver nanoparticles and copper nanoparticles induced in PLA; the mechanical characteristics are studies such that they can be used for any medical applications with need for antimicrobial activity. Each test had a specimen built according to the ASTM standards and the respective mechanical properties are calculated for AgNP PLA and CuNP PLA.

3.1 Filament Preparation

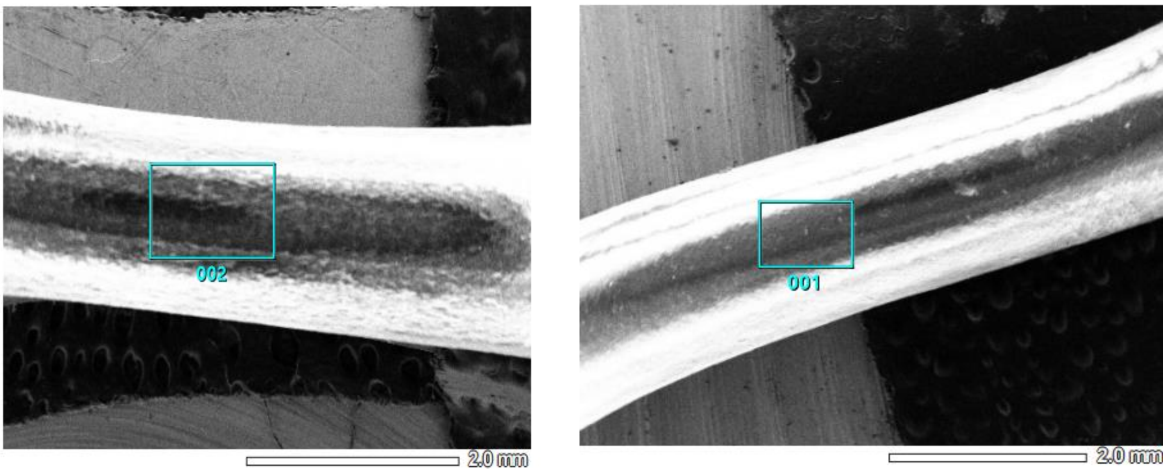
PLA, polylactic acid is a biodegradable thermoplastic monomer. For this type of materials, a plastic extrusion machine called as filament extruder is used to prepare filaments that are put in spool for fused deposition modeling purpose. Below figure shows the filament extruder used for the purpose of extruding both metal nanoparticle induced PLA.

Figure 6: Filament extrusion setup

The principle of the filament works on melt-mixing, the raw plastic granules are melted in a feed hopper with additives, in this experiment, the additives are silver nanoparticles and copper nanoparticles. Two spools are prepared with respective metal nanocomposites individually. The filament extruded goes through a nozzle so that filament close to diameter can be obtained, the filament we used has a diameter of 1.75mm. the wet filament after melting and extrusion goes through a water bath or a hexane bath, where it cools down, using rollers the ultimate diameter is obtained and then the filament is wound into a spool. The above Figure 6 shows a schematic representation of filament extrusion.

4. SCANNING ELECTRON MICROSCOPY (SEM)

Scanning electron microscopy (SEM) employs light emission energy electrons to produce a variety of signals on the outside of strong instances. The data uncovered by electron-test cooperation includes exterior morphology (surface), substance organization, and the glasslike design and direction of the materials that make up the sample. In numerous applications, infrared technology is used. In many applications, data is gathered over a predetermined area of the outside of the device, and a two-dimensional image is formed that depicts spatial variations in these qualities. In a checking mode, regions ranging in width from 1 cm to 5 microns can be scanned using standard SEM techniques (amplification going from 20X to roughly 30,000X, spatial goal of 50 to 100 nm). The SEM results after analyzing AgNP+PLA and CuNP+PLA are as shown in Figure 7(a) and Figure 7(b). The nano particles of respective materials are aggregated in between the filament. The particles at the light scattering are the nano particles that we required. If we need clearer and further results, we need to go for the sonification process.



(a) (b)

Figure 7: (a) SEM image of AgNP+ PLA filament; (b) SEM image of CuNP+PLA filament

5. RESULTS AND DICUSSION

5.1 Tensile test:

The testing was performed according to ASTM standard on FIE Make Universal Testing Machine (UTES-40) machine of 40KN capacity which is shown in the below Figure 8 (a). The width of the specimen was 13.02 mm and thickness were 4mm for silver nanoparticle induced PLA specimen. While, the width was 13.1mm and thickness was 4.05mm for copper nanoparticle induced PLA specimen. Within 5 mm of each end of the gauge length, the width and thickness of each specimen were measured at the center. Specimens were carefully put in the grips of the UTES Ultimate testing machine, with the long axis of the specimen and the grips aligned with an imaginary line connecting the points of the specimen and the grips as shown in the Figure 8 (b) below. Prior to testing the specimens were heated till 80°C for 45 min.

The grips are attached to the machine. The grips are tightened enough to not break the specimen and the gauge length is observed between the grips. An extensometer that has been calibrated is attached to specimen at gage length. crosshead speed is set and during the test the software that is built-in records the data points of load and displacement from extensometer and plots the load-displacement curve of specimen under tensile test. Tensile strength, elongation at break and stress-strain curves can be calibrated and recorded.

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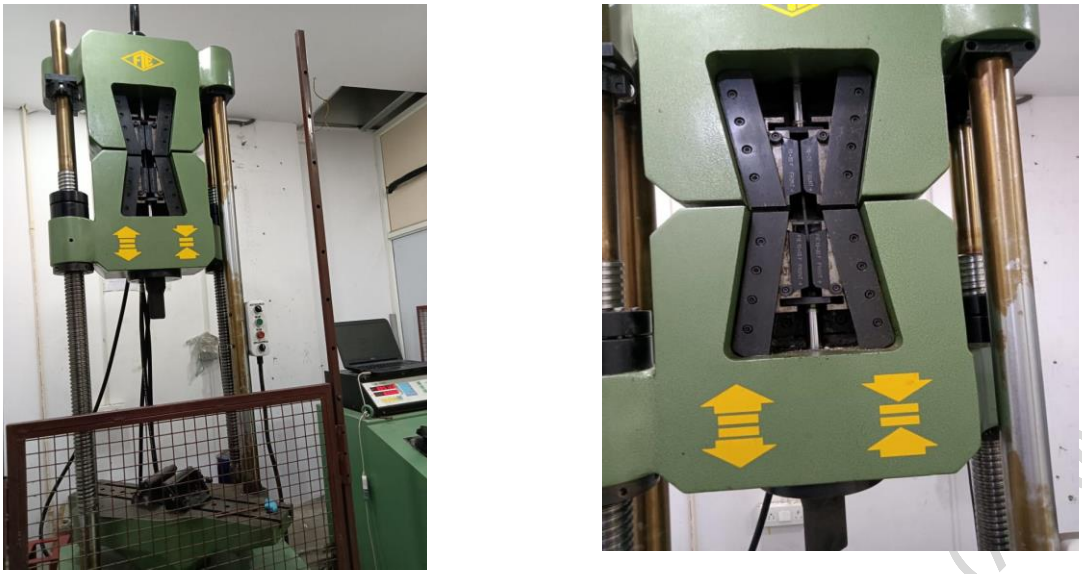


(a) (b)

Figure 8: (a) Mechanical test conducted on UTES 40KN (tensile test); (b) close up of grips used in tensile test

The head of the UTM machine as shown in the Figure 8 (a) has its upper part stationary, while the lower part moves up and down and thus applying the tensile test on the specimen. Tensile test specimen according to ASTM D638 type 1 standard is shown below in Figure 9 and Table 1.

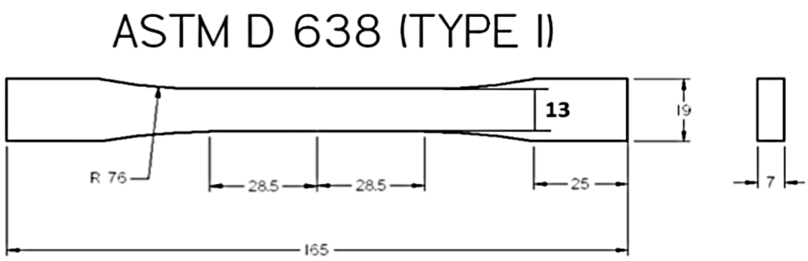


Figure 9: Tensile test specimen according to ASTM D638 type 1 standard

Table 1: Dimensions recommended according to ASTM D638 (Type1)

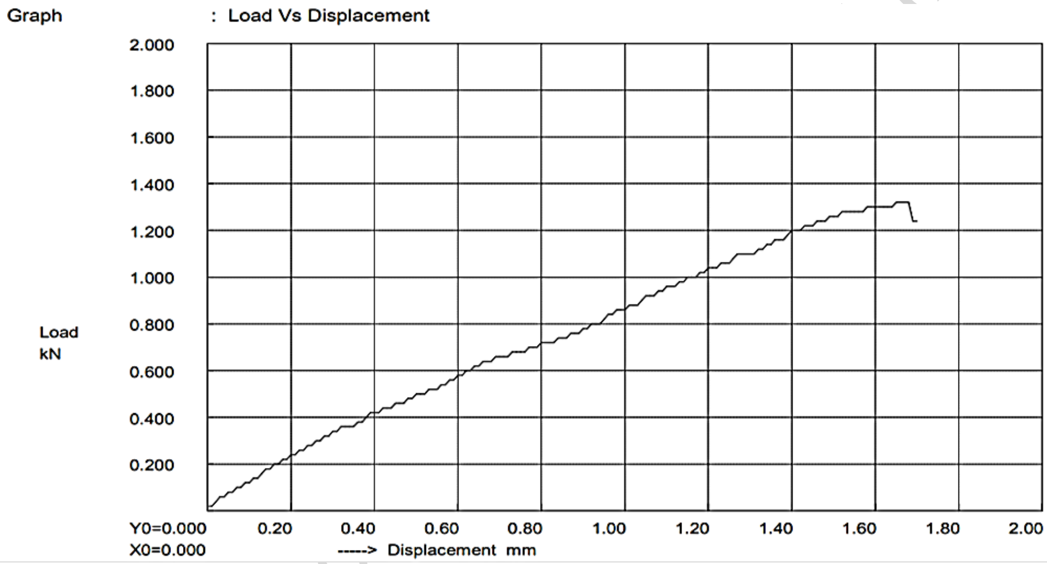
Size	**Full length(mm), l_3**	**Parallel length (mm), l_2**	**Gauge length (mm), l_1**	**Parallel section width (mm), b_1**	**Thickness (mm), h**	**Grip section width (mm), b_2**	**Distance between grips (mm)**
Type 1	165	57	50	13	7 or less (Recommended 3.2±0.4 mm)	19	115

For both the specimens, infill density was 80% and layer height was 180 microns. From standardized test the load vs displacement graph obtained is displayed below in Figure 10(a) and Figure 10(b) and test results of the specimens AgNP+PLA and CuNP+PLA as shown in the Table 2 predict that with higher gauge length the ultimate tensile strength (UTS) varies

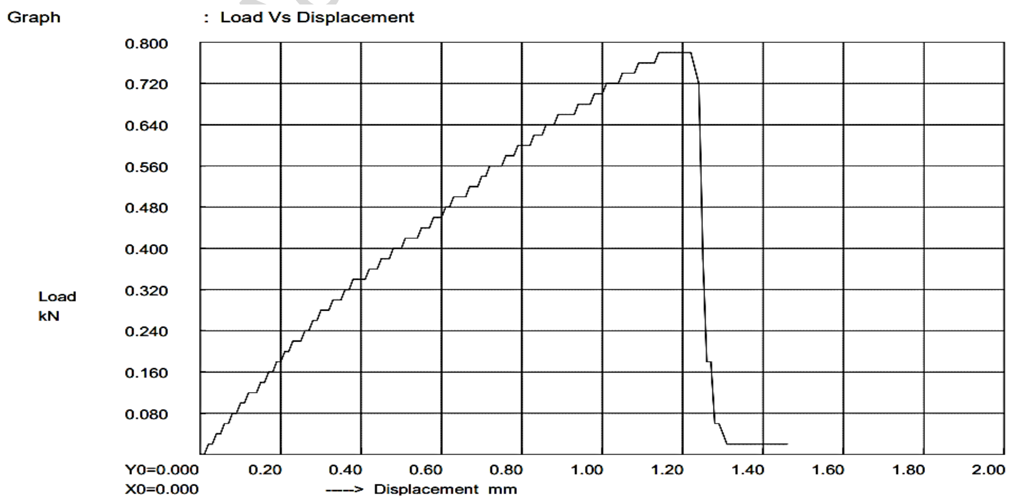
Table 2: Tensile test results

Specimen	Ultimate Load (kN)	Ultimate Tensile Strength (Mpa)	Area (mm^2)	Gauge Length (mm)
AgNP+PLA	1.320	25.346	52.080	50
CuNP+PLA	0.780	14.702	53.005	50

From the data points of load and the values of extensometer obtained during the tensile test, load vs displacement graphs are plotted for both AgNP+PLA and CuNP+PLA specimens.



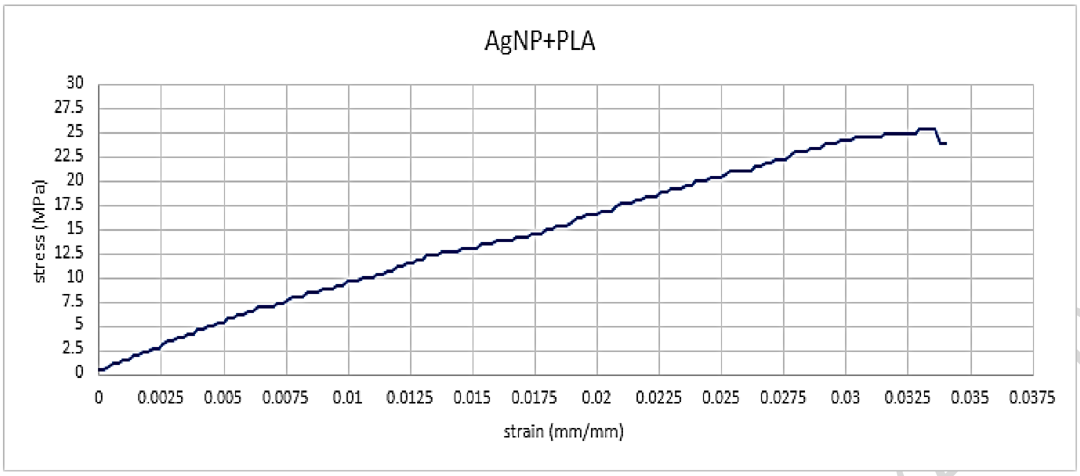
(a)



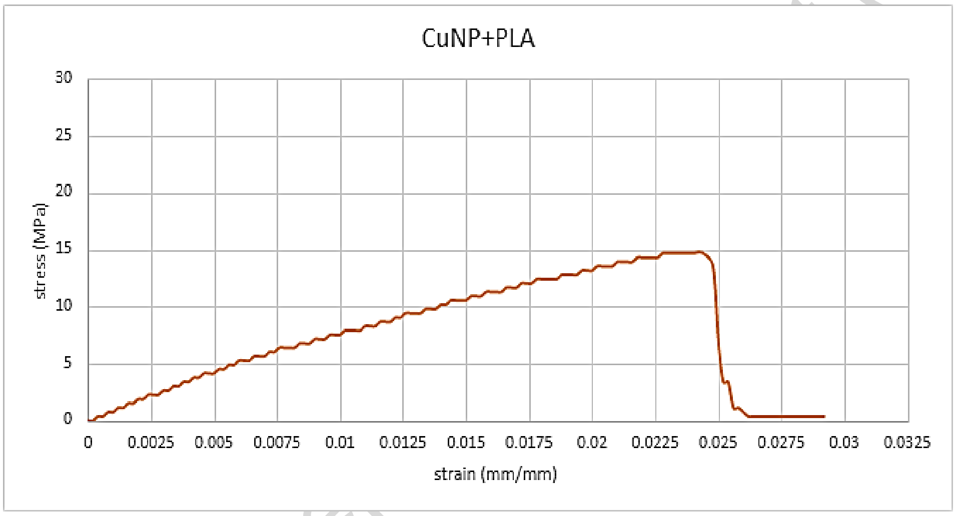
(b)

Figure 10: load vs displacement graphs (a) AgNP+PLA (b) CuNP+PLA

5.1.2 Statistical results - Tensile test



(a)



(b)

Figure 11: stress vs strain curves (a) silver nanoparticle induced PLA (b) copper nanoparticle induced PLA

For tensile test on silver nanoparticles with PLA and copper nanoparticles with PLA, from load vs displacement graphs, a stress vs strain graph is obtained as shown from the Figure 11 above. It can be observed that the silver nanoparticle induced PLA has more tensile strength than copper nanoparticle induced PLA. Stresses and strains are calculated using traditional formulas. The area under stress-strain curve is toughness, we can also say that toughness of AgNP+PLA is more than Cu+PLA composite. The ultimate tensile strength of silver nanoparticle induced PLA is more than that of copper nanoparticles induced PLA. Below bar graph shown in Figure 12 is a pictorial representation of ultimate tensile strength of both metal nanoparticles induced with PLA.

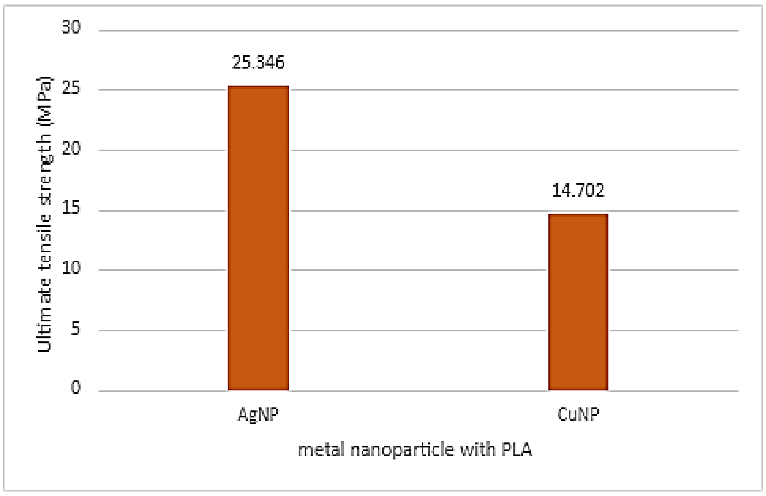


Figure 12: Tensile strengths of both metal nanoparticles induced in PLA

5.2 Compression Test

Compressive testing is carried out in the same manner as tensile testing, using the same apparatus. Axial compression testing is utilized to predict the material response to different crushing loads, as well as to measure the material's plastic flow behavior. The below Figure 13 shows the compression test conducted on silver nanoparticle induced PLA specimen.

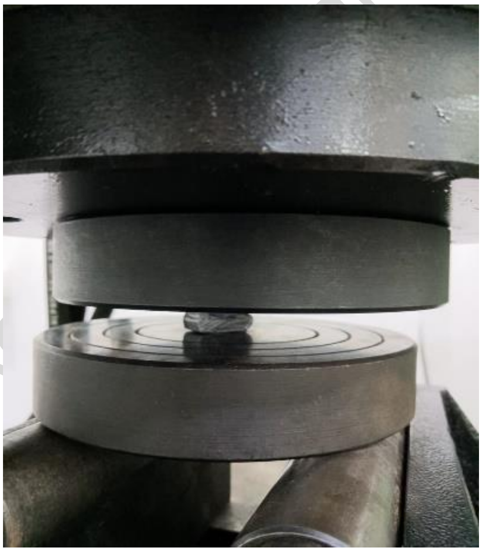


Figure 13: Compression test (ASTM D695) of AgNP+PLA specimen

The testing and analysis results can help in determining the applications of materials. According to ASTM D695 the specimen is 3D printed, below Figure 14 represents the dimensions as per ASTM standards.

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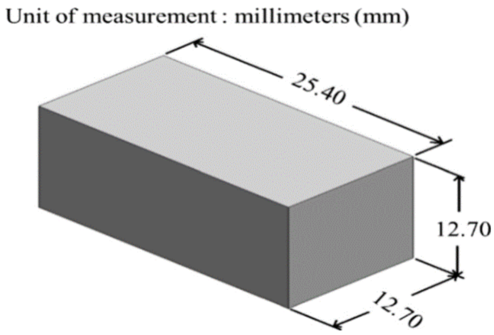


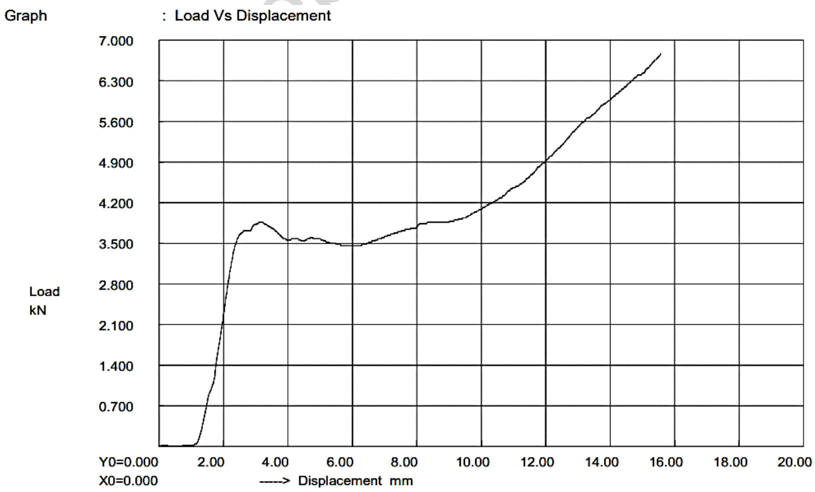
Figure 14: ASTM D695 specimen for compressive test

In compression test the anvil moves towards the stationary head such that forces act on the specimen component and from the standardized testing software we can get the fata points of the load applied and the compression in the specimen, based on this load vs displacement graphs are drawn for both AgNP+PLA and CuNP+PLA materials, as shown in below Figure 15(a) and Figure 15 (b) and Table 3 which represents the results obtained after the compression tests

Table 3: Compression test results

Specimen	Ultimate Load (kN)	Ultimate Compressive Strength (MPa)	Area (mm^2)	Height (mm)
AgNP+PLA	6.760	56.766	119.085	50
CuNP+PLA	2.760	22.885	120.602	50

For both the specimens, infill density was 80% and layer height was 180 microns. From standardized test the load vs displacement graph obtained is displayed below.



(a)

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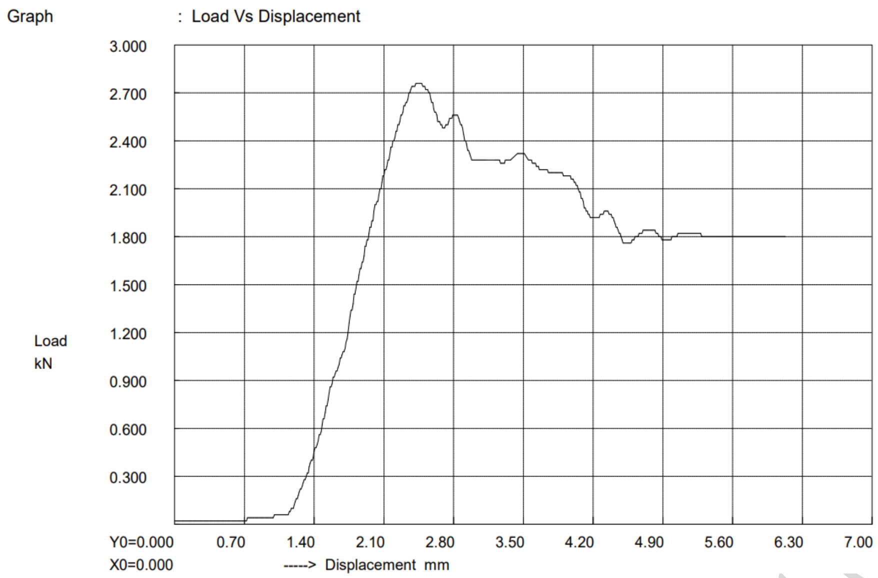
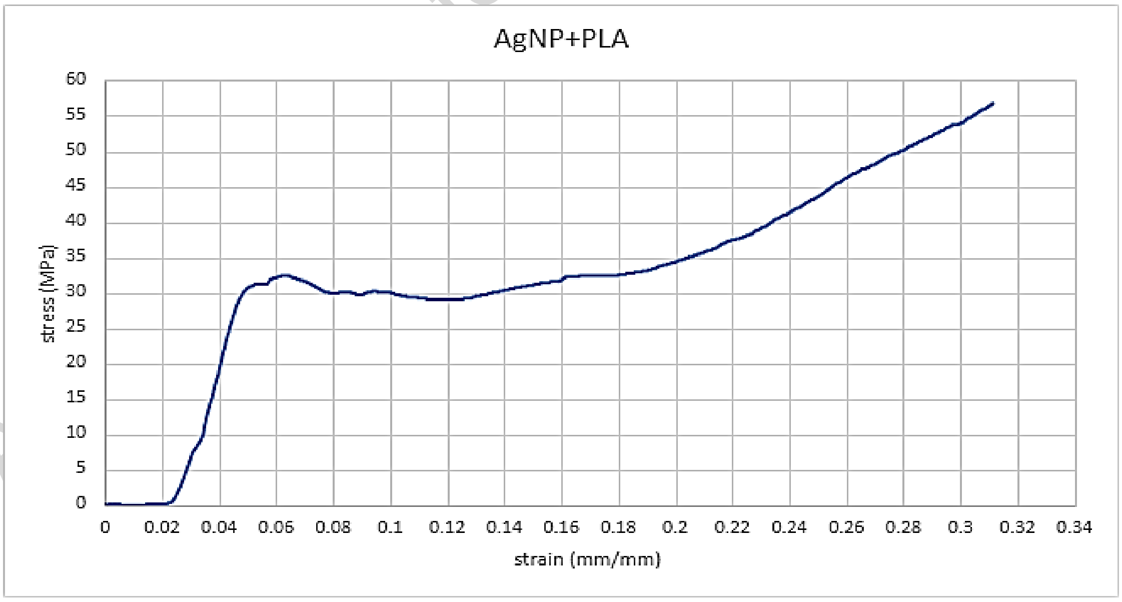


(b)

Figure 15 : load vs displacement graphs (a) AgNP+PLA (b) CuNP+PLA

5.2.1 Statistical results - Compression Test

The test was conducted according to ASTM D695 standards on the same machine as tensile test. From the load vs displacement graph obtained from the standardized test and the data points we get the stress vs strain graph. From the below Figure 16(a) and 16(b) we can observe that the compressive strength of silver nanoparticle induced PLA is more than that of copper nanoparticle induced PLA.



(a)

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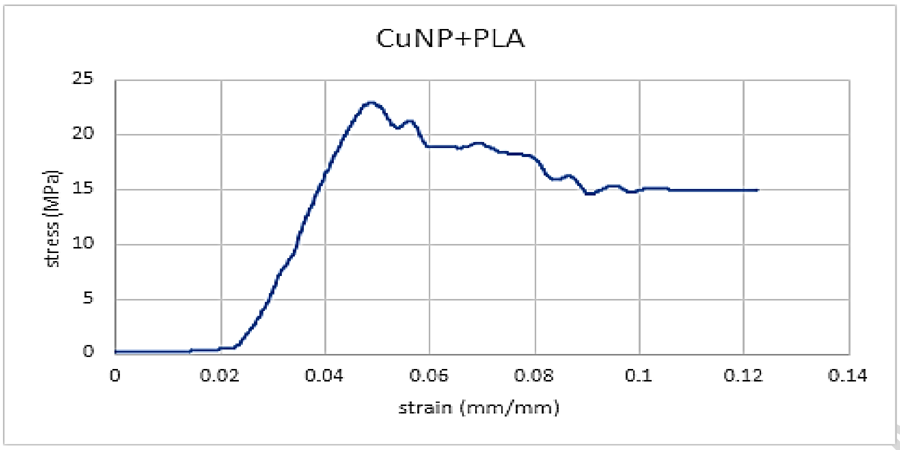

(b)

Figure 16: stress vs strain curves (a) silver nanoparticles induced PLA (b) copper nanoparticles induced PLA

The ultimate compressive strength of silver nanoparticles induced PLA is 32.41 MPa while that of copper nanoparticles induced PLA is 22.9MPa, the pictorial representation is given in below Figure 17.

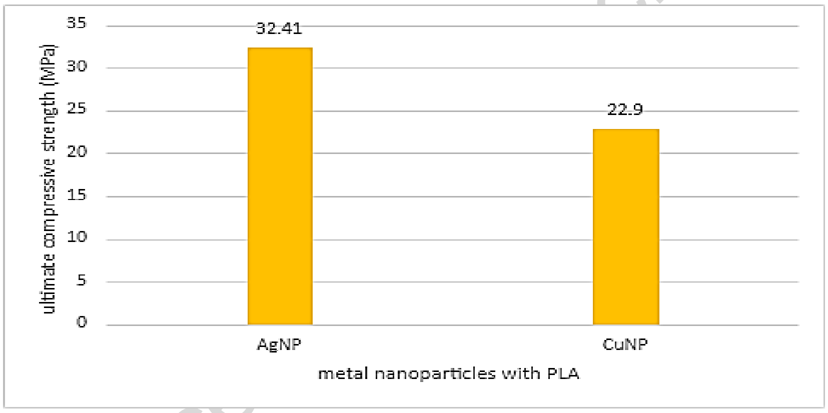

Figure 17: Pictorial representation

5.3 Flexural Test

Bending fixtures with two and three points are often utilized. As indicated in Figure 18, below the grips in FIE UTES machine is replaced by fixtures.

Figure 18: Flexural test (ASTM D790) of AgNP+PLA

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The dimensions of specimen are taken according to ASTM D790, the specimen Figure 19 is shown below and unlike for tension and compression, the data points were not collected instead the ultimate flexural strength for both specimens were obtained.

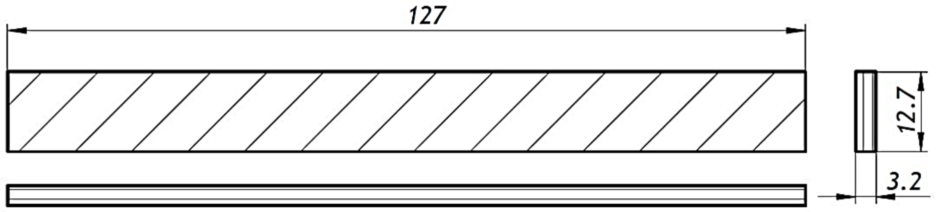


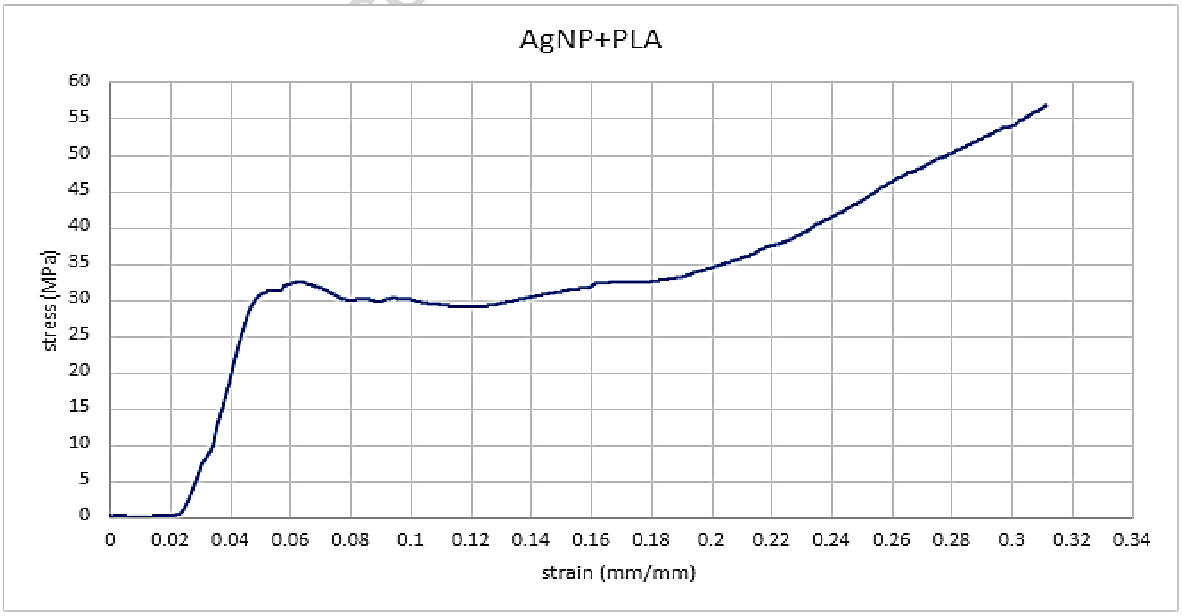
Figure 19: Flexural test specimen (ASTM D790)

For both the specimens, infill density was 80% and layer height was 180 microns. The flexural test results obtained are tabulated below in the Table 4.

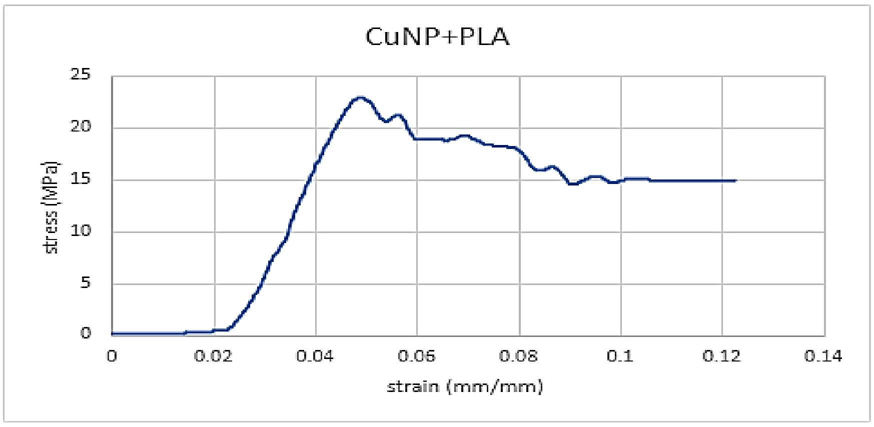
Table 4: Flexural test results

Specimen	**Width, B (mm)**	**Thickness, H (mm)**	**Ultimate Flexural Strength (MPa)**
AgNP+PLA	12.97	1.42	53.05
CuNP+PLA	13.05	1.46	41.79

The experiments were performed in according to ASTM D695 standards on the same machine as tensile test. From the load vs displacement graph obtained from the standardized test and the data points we get the stress vs strain graph. From the below Figure 20(a) and Figure 20(b) we can observe that the compressive strength of silver nanoparticle induced PLA is more than that of copper nanoparticle induced PLA.



(a)



(b)

Figure 20: stress vs strain curves (a) silver nanoparticles induced PLA (b) copper nanoparticles induced PLA

The Ultimate compressive strength of silver nanoparticles induced PLA is 32.41 MPa while that of copper nanoparticles induced PLA is 22.9MPa, the pictorial representation of the above data is represented in Figure 21 below.

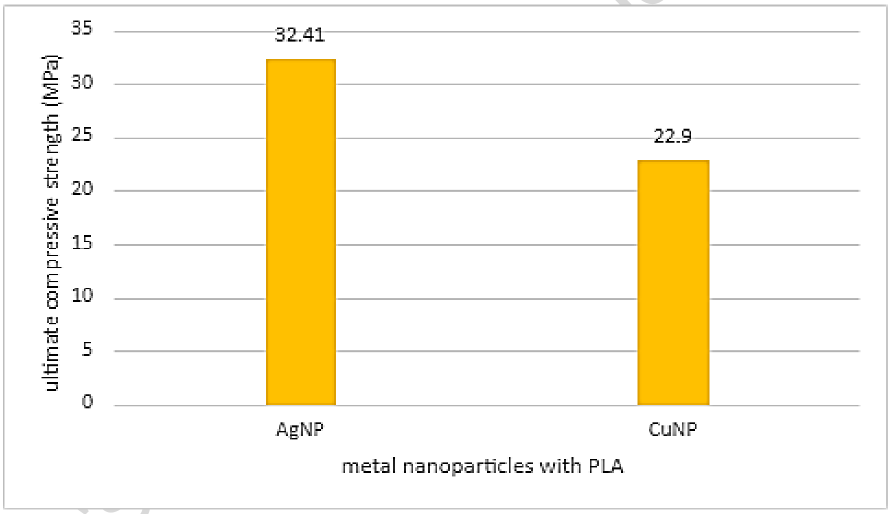


Figure 21: Pictorial representation

5.3.1 Statistical data - Flexural Test

A 3-point bending test was used to find the flexural strength, it is basically the amount of flexing the material can take before permanent deformation or braking. The flexural strength is obtained for both the specimens but unlike for compression and tension test, standardized testing is not available. The pictorial representation of flexural strength is given in below Figure 23, After the observation from the tests, flexural strength of silver nanoparticle specimen was found to be more than copper nanoparticle specimen.

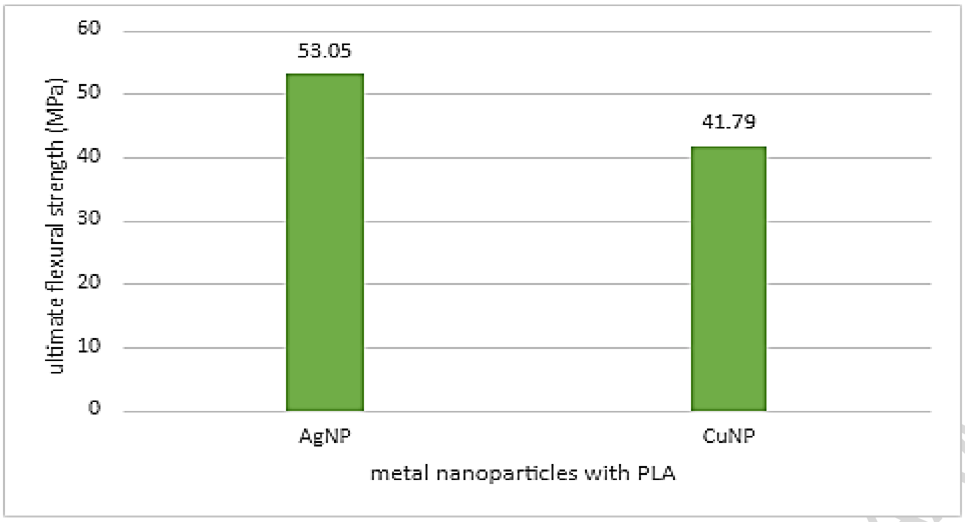


Figure 23: ultimate flexural strength of both metal nanoparticles induced PLA

6. CONCLUSION

The subsequent conclusions that were obtained based on the results are that the specimens produced the mechanical strength properties of the metal nanoparticles induced PLA, silver nanoparticle when induced into PLA has given tensile strength, compressive strength and flexural strength compared to copper nanoparticle induced PLA. For the same size of silver and copper nanoparticles, when fill density was 80%, with a layer thickness of 180 microns, we got the ultimate tensile strength as 25.35MPa for silver nanoparticle and 14.70MPa for copper nanoparticle. The strength of silver nanoparticle specimen was 72.4% more than the copper nanoparticle specimen. In the same conditions of printing as tensile specimen, the compression specimens were printed and according to the compression test done, the ultimate strength for silver nanoparticle composite was 32.41MPa and for copper nanoparticle specimen was 22.90MPa. The compressive strength of silver specimen was observed to be 41.5% more than that of copper specimen. The flexural test was also carried out at 80% fill density and 180 microns thickness of layer, according to the three-point bending test, for silver nanoparticle specimen, the flexural strength was observed to be 53.05MPa and for copper nanoparticle specimen, the flexural strength was 41.79MPa, the silver specimen's strength was 26.94% more than the flexural strength of copper specimen. For all the mechanical properties that were studied, it can be noted that silver had more strength (mechanical, compressive and flexural) than copper nanoparticle specimen.

FUNDING

The authors have not disclosed any funding.

CONFLICT OF INTEREST

The authors declare that they have no financial or non-financial competing interests.

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