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Optimization of Hot Press Compression Molding and Fabrication of Poly Lactic Acid (PLA) Luffa Biocomposites for Biomedical Applications

[1]Akshay Kakar, [2]Elammaran Jayamani, [3]Soon Kok Heng, [4]Muhammad Khusairy Bin Bakri, [5]Sinin Hamdan

[1,2,3,4]Faculty of Engineering, Computing and Science, Swinburne University of Technology Sarawak Campus, Jalan Simpang Tiga, 93350, Kuching, Sarawak, Malaysia
[5]Faculty of Engineering, University Malaysia Sarawak, 94300 Kota Samarahan, Kuching, Sarawak, Malaysia

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ABSTRACT

Biopolymer composites for tensile testing were fabricated according to the American Society for Testing Materials, ASTM D638-10, using poly lactic acid (PLA) as the matrix material and varying volume percentage of heat treated and untreated luffa fiber as the reinforcement material. To achieve the composite materials with the highest tensile strength, optimization of the use of hot press machine for fabrication of composite specimens was given high importance. While optimizing the use of hot press machine, various parameters were considered. These parameters included processing temperature, processing time and cooling time. Combinations of varying magnitudes of these parameters were used to find the optimum processing method. The optimized method was then used to produce PLA-luffa composites. The fiber-matrix interface adhesion was studied using scanning electron microscope (SEM). The results show that composites made with heat treated fibers have higher tensile strength and better interfacial adhesion when compared with the tensile strength of composites made with untreated fiber. Heat treated fibers showed a remarkable improvement in the tensile strength of the biocomposites, which may be used for a variety of applications in the orthopedic field.

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INTRODUCTION

In the recent years, the development of polymer matrix composite materials have steered technological developments across a wide range of applications in the biomedical industry (Scholz *et al*., 2011). In the field of orthopedics, fiber reinforced polymer composites are the most widely used multiphase materials. In addition, most of today`s upper and lower limb prostheses are now made from composites with underlying polymer matrix (Jenkins and de Carvalho, 1977). Composites are a combination of two or more discrete components. One component is called matrix, whereas the other component, referred to as reinforcement, is intermixed within this matrix, providing changed and improved properties to the material as a whole (Sen, 2004). Composites typically possess a superior strength to weight characteristics compared to monolithic materials and offer excellent biocompatibility. Therefore, they possess promising properties for both, soft and hard tissue applications and the design of prostheses in biomedical applications (Scholz *et al*., 2011). Bones and teeth are good examples of hard tissues, whereas skin, cartilage, ligaments and blood vessels are examples soft tissues. Generally, the hard tissues are stiffer and stronger than the soft tissues. Biocompatibility of materials is the indication of the material's ability to exist and perform in harmony with living organisms. Specifically, one distinguishes between the physical, biological and chemical suitability of a material and its compatibility in terms of mechanical properties such as optimal load transmission, stiffness, and strength at the implant or tissue interface (Wintermantel *et al*., 2001). PLA has been widely studied for use in medical applications because of its bioresorbability and biocompatible properties in the human body. Biomaterials are organic materials which may exist in nature or may be synthesized in laboratory, using naturally existing materials. They are used for supplementing, directing, or replacing the functions of living tissues in the human body (Black, 2005). The main reported examples on medical or biomedical products are fracture fixation devices like delivery systems, sutures, screws and

Corresponding Author: Akshay Kakar, Faculty of Engineering, Computing and Sciences, 93350, Kuching, Sarawak, Malaysia
Tel: +60 82 416353 E-mail: akakar@swinburne.edu.my

micro-titration plates (Doi and Steinbuchel, 2002). Several researchers proposed a variety of polymer composite materials for bone plate applications (Tayton *et al*., 1982). Bone plates were then classified into three categories: fully resorbable, partially resorbable and non resorbable.

The polymers such as poly glycolic acid (PGA) and poly lactic acid (PLA) resorb or degrade upon implantation into the body (Majola *et al*., 1991). These bioresorbable polymers were found to loose most of their mechanical properties within a few weeks. These polymers are either very brittle or very flexible for safe clinical use that involve load bearing applications. One of the advantages often sighted for resorbable composite prostheses is that they need not be removed with a second operative procedure, as is recommended with metallic or non-resorbable composite implants (Ramakrishna *et al*., 2001). Choueka *et al*., (1995) and Tormala *et al*., (1991) proposed fully resorbable composites by reinforcing resorbable matrices with resorbable fibers (poly-L-lactic acid) fibers and calcium phosphate based glass fibers. The degradation rate of resorbable composites may be engineered by adding, for example, coupling agents, fiber treatments, or coatings. The processing and characterization of phosphate glass fiber, a completely resorbable and bioactive material, for tissue engineering was studied by Ahmed *et al*., (2004). Natural fiber composites may be used for biomedical applications for bone and tissues repair and reconstruction (Dhandayuthapani *et al*., 2011). Innovations in the composite material design and fabrication processes are raising the possibility of realizing implants with improved performance by using plant fibers based biocomposites (Namvar *et al*., 2014). Usage of biomaterials obtained from sustainable materials has been significantly rising due to increasing prices of petroleum products and the demand for environmentally friendly and sustainable biomedical devices. Natural fiber reinforced polymer composite materials, which are less rigid than metals, may be good alternatives because their mechanical properties are closer to the bones' mechanical properties. It was found that biocomposites help to avoid stress shielding and increase bone remodeling (Ali *et al*., 1990). Studies have been conducted recently on bio-fibers reinforced poly lactic acid biocomposites to discover their mechanical properties, thermal properties and biodegradability. These biocomposites include: bamboo fiber – PLA biocomposites (Lee and Wang, 2006), corn starch – PLA biocomposites (Ohkita and Lee, 2006) and microcrystalline cellulose – PLA bicomposites (Mathew *et al*., 2005). These studies show the development of biocomposites used in the biomaterials industry. In this research, we aim to develop new alternative biomaterials known as biodegradable implants with PLA as the matrix material and luffa fiber as the reinforcing phase. Tensile strengths of the biocomposites were evaluated between composites that were fabricated using heat treated and untreated luffa fibers. Interface before and after heat treatment of fibers and matrixes were studied using the Scanning Electron Microscope (SEM).

Fig. 1: Luffa skeleton.

Methodology:

Materials:

Poly lactide, or poly lactic acid, is one of the most popular biopolymers. Poly lactic acid is polyester made from lactic acid that can be derived from natural sources such as corn (Sujaritjun *et al*., 2013). The poly lactic acid (PLA), usually found in crystal form, is brittle and biologically degradable. It is produced from lactic acid (2-hydroxypropane acid), which, when heated, forms a cyclic compound called di-lactide due to the splitting of water molecules in a condensation polymerization reaction (Morrison *et al*., 1986). A poly-lactic-acid with the trade name "AI - 1001" has the density of 1.40 g/cm^3, the melting point of 150°C, the melt index of 10 to 12 g/10 min, and the elongation break at 2%. This poly-lactic-acid was supplied by Dalian Lejia Chemical Co. Ltd. (China) and was used as the matrix material in this research.

Luffa is a tropical fibrous vegetable. When the vegetable is dried after it fully ripens, it leaves behind only the fibrous skeleton and seeds, as shown in Fig. 1. The seeds are removed by simply shaking the fibrous skeleton. Luffa fiber skeleton is usually used as a scrub. Its chemical composition may vary depending on its origin, climatic conditions under which it was grown, soil in which it was grown and few other biological factors. Its density falls within

the range of 0.82 g/cm^3 to 0.92 g/cm^3 (Satyanarayana *et al*., 2007; Tanobe *et al*., 2005). Its lignin content may vary from 10 % to 23 %, cellulose varies from 55 % to 90 % (Satyanarayana *et al*., 2007), hemicellulose content may range from 8 % to 22 % (Satyanarayana *et al*., 2007), ash 0.4 % and extractives of about 3.2 % (Satyanarayana *et al*., 2007). The luffa fiber skeleton was obtained from a local market in Kuching, Sarawak, Malaysia. The skeleton was then cut to turn it into short fibers. Half the amount of fibers used in this study underwent heat treatment. In this study, both, heat treated and untreated fibers were used.

Methods:

Physical Treatment:

The fibers were chopped to an average length of 1mm to 10mm. They were then heat-treated in the Ecocell EC55 oven (MMM Medcenter Einrichtungen, GmbH (Germany)), with natural air convection at the temperature of 160 °C for 10 minutes. Luffa fibers were subjected to heat treatment in order to improve the interfacial adhesion between the luffa fiber and the PLA matrix. The natural fiber and polymer matrix interface adhesion plays an important role in the physical and mechanical properties of composite materials. Heat treatment is a physical method that does not change the chemical composition of the fibers. Heat treatments of natural fibers are based on the fact that hemicellulose has a lower decomposition temperature than cellulose. Strongly hydrophilic materials such as hemicelluloses can therefore be removed from the fibers by heating. This changes the molecular surface free energy and the structure of the fibers, and improves the interfacial adhesion between the hydrophilic fibers and the hydrophobic polymers (Huang *et al*., 2013).

Design and Fabrication of the Frame:

The frame that was designed and fabricated for this study was in accordance with the American Society for Testing Materials (ASTM D638-10, 2010). The frame was made of stainless steel SAE 304, as it is known to retain strength at high temperatures and possess good heat dissipation characteristics. This frame was designed while keeping the cooling rate in mind. No male-female mold was used, as it would increase the thickness of the entire mold, causing it to store more heat energy. Higher the heat energy stored in the mold, lower would be the cooling rate. Moreover, a male-female mold would make removing the samples a hassle and would require extra pressure on the samples during extraction. This would damage the samples before conducting the tests. Hence, the ease of use, the sample extraction process and high cooling rate were optimized through this design. An image of the frame used is shown in Fig. 2.

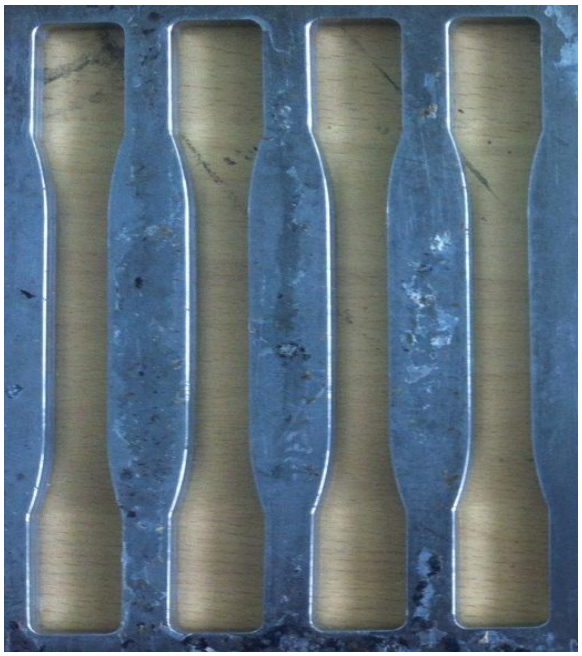

Fig. 2: Tensile test frame made from stainless steel SAE 304.

Fabrication of Composites:

Injection molding and extrusion are the more popular methods of fabricating PLA composites. This research has contributed to the possibility of using compression molding to fabricate composites. According to Garkhail *et al*., (1999), compression molding produces composites with better mechanical properties when compared with composites fabricated using injection molding. In this study, the machine used for compression molding was the 30

Ton Hydraulic Hot Press LS-22071, supplied by Lotus Scientific, Malaysia.

A number of experiments were conducted which involved variation in processing temperature, heating procedure, processing time and cooling time of PLA samples to optimize the use of hot press machine. The processing temperature is the temperature at which the machine was set, to melt the poly-lactic-acid and fabricate the composites. The processing time is the duration for which the poly-lactic-acid was heated at the processing temperature. The cooling time is the time taken for the poly-lactic-acid and the frame to drop in temperature from the processing temperature to the room temperature (initial temperature). The temperature varied from 150 °C to 170 °C, processing time varied from 15 minutes to 45 minutes and cooling time varied from 4 hours to 15 minutes. The two procedures used to heat the pure PLA samples included: heating machine from room temperature to processing temperature with the frame in the machine and preheating the machine to the processing temperature before placing the frame in the machine.

For each of the processing temperature, composites were fabricated with all combinations of processing time (15 minutes, 30 minutes and 45 minutes) and cooling time (15 minutes, 1 hour and 4 hours), while heating slowly from room temperature to the selected processing temperature. On testing these composites, the sample with the highest tensile strength was identified. This method was then used in the rapid heating method, where the hot press was preheated to the processing temperature. Samples that were heated slowly had higher tensile strengths compared with the tensile strengths of the samples that were fabricated in the preheated machine.

Occurrence of bubbles in the composite was eliminated by placing baking paper between the plates and the steel frame. Usage of baking paper allowed lower cooling time, as the samples in the steel frame could be directly cooled under air. Without the baking paper, the top and bottom plates had to be cooled as well, because the PLA would stick to the plates. The samples attained the highest tensile strength when heated slowly from room temperature to 160 °C, with processing time of 30 minutes and cooling time of 15 minutes.

The optimized procedure was then used to fabricate PLA samples and PLA luffa composites with 5, 10, 15 and 20 volume percentages, for tensile testing. Baking paper was placed on the base plate, over which the steel frame was positioned. One layer of PLA pellets was laid in the frame. Based on the density of the fiber, its mass equivalent of the volume percentage was calculated. The calculated mass of fiber was then paced on the first layer of PLA pellets. This layer of fiber was then topped with another layer of PLA pellets. An additional baking paper was placed on top of the frame, which was topped by the upper plate. The frame was then placed in the hot press machine and was heated from 25 °C to 160 °C. Once the machine reached 160 °C, the temperature was maintained for 30 minutes. After 30 minutes, the frame was removed from between the top and bottom steel plates and air cooled to room temperature within 15 minutes.

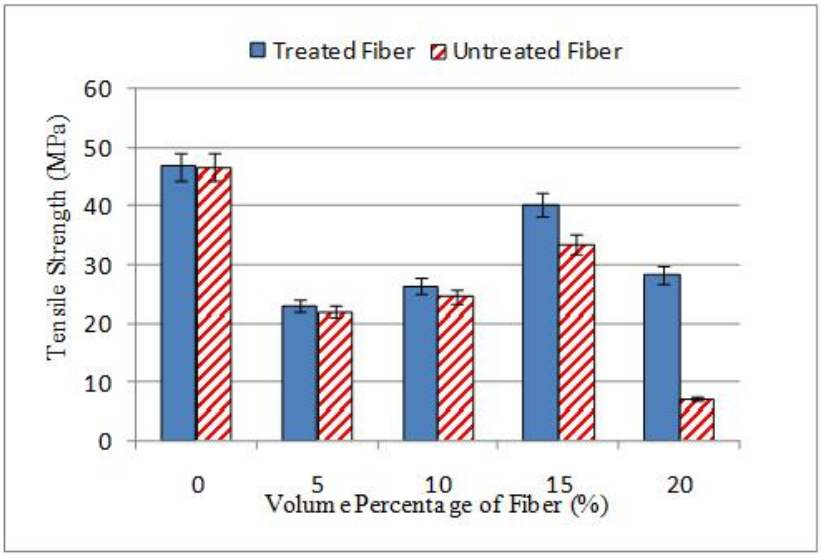

Fig. 3: Tensile strengths of the pure PLA sample, and treated and untreated fiber composites.

Results:

The tensile tests conducted during this study were carried out on LS-28011-50 Universal Testing Machine (UTM) (T-Machine Technology Co., LTD, Taiwan). The Fig. 3 shows the tensile strength of the composites with respect to the volume percentage of fiber present in them. Pure PLA had the highest tensile strength (46.8 MPa). With the addition of 5 % luffa fiber, the tensile strength drops for both, treated (23.05 MPa) and untreated (22.04 MPa) fiber

composites. With an increase in the fiber percentage to 10 %, a slight increase in the tensile strengths of treated (26.37 MPa) and untreated (24.53 MPa) was observed. When the fiber was increased to 15 %, a significant increase in the tensile strengths of treated (40.20 MPa) and untreated (33.42 MPa) fiber composites was observed. However, at 20 % fiber volume, the tensile strength of treated fiber composite (28.27 MPa) dropped, but was higher than that of 10 % treated fiber composite. Whereas, the tensile strength of the untreated fiber composite (7.18 MPa) reduced drastically and dropped lower than that of 5% untreated fiber composite. Generally, the treated fiber composites demonstrated higher tensile strength when compared with the untreated fiber composites of the same fiber percentage. The highest tensile strength of luffa fiber composite was found to be at 15 %, with treated fiber.

Discussion:

Optimization:

During the optimization process of the machine, it was found that PLA samples with processing temperature of 150 °C did not melt completely. This left the pellets in the middle of the sample partially melted and partially in the pellet form. This made the samples extremely weak, causing them to break while extracting them from the frame. All samples that had cooling time for 4 hours, cracked during extraction, making them unfit for testing. On the other extreme were all the samples that were processed at the temperature of 170°C for 45 minutes. These samples were extremely brittle, causing them too to break while extracting from the frame. Similar issue occurred while using the pre-heating technique. These samples broke during extraction due to presence of excessive amount of bubbles. The sudden rise in the temperature caused some amount of the PLA to vaporize. Since the frame was shut tight under the pressure of 5 MPa, the vapor could not escape, leaving back packets of bubbles throughout the samples. These bubbles made the samples rather weak, causing them to break under the slightest force used to retrieve them. The samples fabricated with the processing temperature of 170 °C with 30 minutes of processing time and 15 minutes of cooling time produced samples with an average tensile strength of 14 MPa. This tensile strength was low mainly due to the multiple bubble packets in the samples. However, samples with cooling time of 1 hour and 4 hours, with processing temperature of 170 °C and 30 minutes of processing time were brittle and had bubbles, making the samples crack and break during extraction from the frame. Samples fabricated with 170 °C, 15 minutes processing time and 15 minutes cooling time had an average tensile strength of 15.75 MPa. All other samples that were heated for 15 minutes at the processing temperature were unfit for testing. Samples that were heated at 160 °C for 45 minutes and cooled in 1 hour broke during extraction. PLA samples produced with 160 °C, 45 minutes of processing time and 15 minutes of cooling time had the average tensile strength of 4 MPa. This was due to the high brittle characteristic of the samples. PLA samples with processing temperature of 160 °C, 30 minutes processing time and 1 hour of cooling time had an average tensile strength of 16.4 MPa. On using the slow heating (from room temperature to processing temperature) method with 160 °C processing temperature, 30 minutes processing time and 15 minutes of cooling time produced the samples with significant improvement in the samples' tensile strength. These samples had the average tensile strength of 46.8 MPa. This drastic improvement in the strength was due to lack of bubbles, increase in the ductility of the samples and the complete melting of pellets, enabling them to form a strong bond throughout the samples.

Large air packets were produced during the cooling process, when the melted PLA sticking to the top plate maintained its position and the rest of the liquid PLA reduced in volume. The gap created was filled with air. The use of baking paper between the frame and the top and bottom plates significantly helped in achieving high cooling rate and avoiding large air packets caused due to the PLA sticking to the plates. The baking paper helped to increase cooling rate, as the frame and samples alone had to be cooled, leaving out the heat from the top and bottom plates.

The two major drawbacks of producing composites using compression molding method are the occurrence of bubbles and high level of brittleness of the samples when not fabricated in the optimized method. These two factors can render the samples redundant. As far as processing temperature and processing time are concerned, these factors depend on the grade of the PLA pellets. Processing temperature should be about 10 °C higher than the melting temperature. If the temperature is much higher than that, it may cause some amount of the PLA to vaporize and leave back bubbles in the sample. If it is heated at the melting temperature, it will take more processing time to melt the pellets, making the sample extremely brittle. Generally, the higher the cooling rate, the stronger and more ductile the PLA samples will be. In this study, air cooling method was used, which provided the average cooling rate of 9 °C/minute. Water quenching was not used during this research, as it would make the frame brittle, causing it to crack or break easily.

Testing:

The pure PLA samples clearly had the highest tensile strength. However, this strength is not considered for the application in the biomedical field, as one of the objectives was to reduce the use of PLA and replace it with recyclable agricultural material such as luffa fiber. This would contribute towards a

better agricultural materials management and simultaneously reduce cost of producing this biomaterial. Hence, only the biocomposite materials were taken into account while considering their viability for biomedical application.

Composites with 5 % treated and untreated fiber showed a drastic drop in the tensile strength. This was due to the lack of uniform distribution of the fibers. The fibers were not enough to cover the entire length of the sample consistently. This caused inconsistency in the load distribution, causing the composite to fail at a low tensile strength. The untreated fiber composite had lower tensile strength than the treated fiber composite due to the poor interface bonding the hydrophobic (PLA) and hydrophilic (untreated fiber) materials.

Composites with 10 % treated and untreated fiber volume showed higher strength than the 5% fiber composites. This was caused by a more consistent distribution of the fibers, providing more even load distribution. The untreated fiber composite had lower interfacial strength, which cause it to have lower tensile strength than treated fiber composites.

Composites with 15 % treated and untreated fiber volume showed the highest tensile strength among the composites. The key factor in this increase was the consistency of fiber distribution. The treated fiber composite had the highest tensile strength among all composites in this study due to the high interface bonding and because there was just enough matrix material to mix completely with the fiber. The main reason for the untreated fiber composite to have significantly lower strength was the lack to strong bond between fiber and matrix.

The composites with 20 % untreated fiber volume were the weakest samples. This was due to the weak bonding between fiber and matrix. Since the fiber was not heat treated, it contained the hydrophilic hemicellulose, which did not bond well with the hydrophobic matrix. Another reason for the drop in the strength was lack of PLA for completely covering and bonding the fibers. Some of the fibers were jutting out of the composite, reducing interface surface area and providing openings, which gave the material lower structural integrity. On the other hand, the treated 20 % fiber volume composite showed second highest tensile strength among the composites. This was due to the strong interface bonding. Although the matrix was not enough to provide extreme strength, the interface bonding was very strong, making this composite stronger than the 10% treated fiber composite. This displays the importance of fiber treatment for better interface bonding.

This phenomenon of increase in tensile strength up to certain percentage of fiber and decrease in the strength on further increase of fiber was also observed by Zou *et al*., (2010), while working with wheat straw and polypropylene and by Li *et al*., (2011), while working with sisal fibers and poly lactide. It was also observed that the difference between the tensile strength of treated and untreated fibers increased with the increase in the fiber volume percent present in the composite. This was a clear indicator of the importance of interface bonding between fibers and matrix. The interface bonding could also be further improved with chemical treatment. However, since this study is focused on the mechanical strength of the composites being viable for biomedical use, it was important not to change the pH value of the fiber. Slightest change in the pH factor of the blood can lead to imbalances in the body. Therefore, this study evaluated the tensile strength of the composites solely with the physical change brought about by the heat treatment.

The optimization of the hot press machine not only made it possible to produce composites with high tensile strength, but it also provided a range of tensile strengths, which may be used based on requirements. These requirements may vary in strength from 40.20 MPa to 7.18 MPa. According to Ramakrishna *et al*., (2001), a hard tissue, cancellous bone, has the tensile strength of 7.4 MPa and a soft tissue, articular cartilage, has the tensile strength of 27.5 MPa. Since the tensile strength of PLA-luffa composites ranged from 7.18 MPa to 40.20 MPa, the appropriate composite may be produced to fit the bone plate for external bone implant. This makes PLA-Luffa composites viable bone plate application for certain hard and soft tissues.

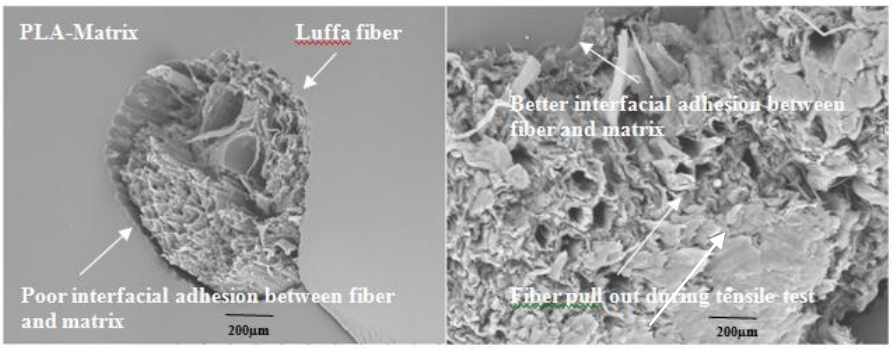

Fig. 4: Morphology of a) untreated, and b) treated PLA-luffa composites.

Morphology:

Fig. 4 shows morphology for the appearance of interfacial adhesion prior to the heat treatment and after heat treatment. In the case of untreated fibers, a considerable gap was observed between matrix and fibers. It shows poor interfacial adhesion. In contrast, when fibers were treated with heat, no gaps were found between matrix and fibers. It shows better interfacial adhesion. The tensile strength of the composites depends on the adhesion between fiber and matrix in addition to strength and modulus of constituents. It was observed from the Fig. 4 that the tensile strength of PLA luffa composites has been increased after fiber treatment. The enhancement of tensile properties of the treated fiber composites may be due to better fiber and matrix adhesion. Certain chemical treatments tend to severely damage the luffa fibers. Although Tanobe *et al*., (2005), found that treating the fibers with NaOH provides beneficial results; this treatment was not conducted because if some amount of NaOH remains in the fibers, it will create pH imbalances when the composite is used as an implant.

Conclusion:

In this study, the use of hot press machine was optimized for compression molding of PLA and PLA-luffa composites to achieve highest tensile strengths. In addition, the effect of fiber volume percentage present in the composites and the effect of heat treatment of fibers were also studied. While optimizing the hot press machine, it was discovered that generally, lower processing time and high cooling rate have beneficial effect on the tensile strength of PLA. The processing temperature is required to be higher than the melting temperature, to reduce the processing time. However, if this temperature is much higher than melting temperature, the samples produced may have bubbles trapped in them. Existence of bubbles in composites (or pure matrix) has the most adverse influence on the strength. Fortunately, bubbles were avoided very easily by using the right processing temperature and by not allowing the matrix to stick to the plates.

On using the optimized method of compression molding of composites it was found that heat treatment of fibers partially removed hemicellulose, reducing the hydrophilic nature of the fiber, which improved fiber-matrix bonding strength. This treatment and the volume percentage of fiber resent in the composite had the most significant influence on the tensile strength of the composites. The enhanced interface bonding provides good strength and a consistent spread of fibers provided uniform distribution of load when placed for tensile testing. It was found that 15 % volume of treated fibers provided the highest tensile strength (40.20 MPa), when compared to other composites in this study. Overloading the composites with fiber had an adverse effect on its tensile strength, as there was not enough matrix to spread and bond with all the fiber strands.

In a nutshell, PLA-luffa fiber composites showed promising tensile test results. These composites may be used for external bone plate application for certain hard and soft tissues. The fiber volume percentage in the composite may vary depending on the required tensile strength, as providing tensile strength for a tissue with relatively low tensile strength may have adverse consequences. Fiber heat treatment, processing procedure, cooling rate and fiber loading may be adjusted to achieve the desired tensile strength of PLA-luffa composites, depending on the application of these composites in the orthopedic field.

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