INVESTIGATION OF PHYSICO-MECHANICAL PROPERTIES OF ARECA-COTTON FIBER REINFORCED POLYPROPYLYNE COMPOSITE

By

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The thesis titled **Investigation of Physico-Mechanical Properties of Areca-Cotton Fiber Reinforced Polypropylene Composite** submitted by **Gourab Kumar Roy**, Student No. 1017082019, Session- October 2017, has been accepted as satisfactory in partial fulfillment of the requirement for the degree of M.Sc. in Industrial and Production Engineering on 30th July, 2023.

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Declaration

It is hereby declared that this thesis or any part of it has not been submitted elsewhere for the award of any degree or diploma.

Gourab Kumar Roy

Gourab Kumar Roy

This work is dedicated
to my loving

Father

&

Mother

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Abstract

The goal of the current research is to fabricate fiber reinforced composite with comparatively easy to get materials, which comes as byproduct and determine the mechanical, physical, chemical and thermal characteristics of a proposed hybrid polymer composite which consist of a polypropylene matrix and a mixture of cotton and areca fibers. Hybrid composites are usually used as a combination of properties of different types of fibers and a polymer matrix. The advantages of natural fibers are their continuous supply, easy and safe handling, and biodegradable nature. This research work investigated the influence of fibers loading, volume fraction of cotton and areca fiber and different percentage of alkali treatment on the aforementioned characteristic of the composites. Composites with volumetric amounts of fibers up to 15% were fabricated. Various tests like, tensile test, flexural test, impact test, hardness test etc. were carried out for mechanical characterization. The thermal stability of the composite was investigated using thermogravimetric analysis (TGA), the crystallinity of the composite was determined using an Xray diffraction (XRD) test, the surface morphology was examined using Scanning electron microscope (SEM), and the functional groups that were present in the composite were investigated using Fourier transform infrared (FTIR). 15 composites and a composite that had not been treated were created using a mixture that was designed by response surface methodology (RSM). Through desirability function, the optimum fabrication combination was identified. The precision of the prediction model was assessed by comparing the predicted value with the experimental value.

Chapter-1

Introduction

The proper disposal of polymer wastes, which are not environmentally friendly, are difficult to biodegrade, and the processes used to break them down release a lot of hazardous chemicals that further hurt the environment, presents many issues in today's society. And the solution to that problem is to use Natural fibers as reinforcements in polymer composites. Natural fibers are getting popularity as reinforcements in polymer composites because of their low cost, fairly good mechanical properties, high strength to weight ratio, non-abrasive eco-friendliness and biodegradable properties make them more suitable as replacement of conventionally used fibers like glass, carbon etc. Cotton fiber is used heavily in textile industries and Areca nut (Betel nut) is eaten with betel leaf. Everyday a lot of waste cotton fibers are generated from textile industries and areca nut fibers are just thrown out as garbage which is a common scenario in Southeast Asia. Both of the fibers have been used as reinforcement in polymer composite individually and in some cases with other fibers like sisal, jute coir, seaweed, maize, flax. But the present work prepares a new hybrid composite of cotton and areca nut fiber with polypropylene matrix which utilizes the cotton waste and thrown out areca fiber at comparatively lower cost than other synthetic or natural fibers. Different fiber loading, fiber length and alkali treatment will be used for comparison between composites and find out the composite with best physico-mechanical properties by using Response surface methodology (RSM), which can determine the interaction between the independent variables and can reduce time and cost by reducing number of trials. The composite can be used in Aircrafts, boats and marine, sporting packaging industries, low-cost housing, automotive industries (Door panel, seat backs, and bumper), sporting equipment (golf shafts, hockey sticks, tennis rackets, surfboards), wind turbine blades, ladder rails, water pipes and various domestic purposes where strength and cost consideration is important [Oladele et al. 2022].

There are a lot of benefits in using composite materials. They are light weight in nature than most metals. Strength to weight ratio of composite can be made higher than conventionally used materials like metals. Most composites can be made in a way that they can resist weather effect and harsh chemical, whereas most metals are susceptible to corrosion by both. They can be manufactured in complicated shapes than most other materials which show their design flexibility as well. A single piece composite can easily replace entire assembly of metal parts. They can have long life with little to no maintenance [Khosravani 2012].

Composites have a number of advantages, but they also have significant drawbacks. Composites can delaminate where they are weaker because they are frequently made of different ply layers laminated together. Composite materials have relatively recent applications, which make them more expensive to produce. Many industrial processes call for high-quality equipment, which is typically hard to find and challenging to maintain and run. Some of the procedures need a lot of time to build and are labor and complexity-intensive. Internal composite fractures can occasionally appear and are difficult to detect without complex examination methods [Thori et al. 2013].

There are two phases of composite materials like continuous phase and dispersed phase. In continuous phase is primarily called matrix which holds the fibers and transfer shear and usually is ductile and less hard. The dispersed phase is embedded in the matrix which is stronger and can resist tensile and compressive loads. Normally fibers or particulate that works as reinforcing agents are part of dispersed phase [Clyne et al. 2019]. Composites can be of three main categories according to type of matrix like ceramic matrix, metal matrix and polymer matrix. A subcategory of composite materials are ceramic matrix composites (CMCs). They are ceramic fiber reinforced ceramic (CFRC) materials made up of ceramic fibers embedded in a ceramic matrix. Any ceramic substance can be used to create the matrix and fibers. The main drawbacks of traditional technical ceramics, including their brittleness, low fracture toughness, and low thermal shock resistance, were addressed by the development of CMC materials. Metal matrix composites (MMCs) are composite materials that have two or more constituent parts, either different metals or other materials. To increase strength and wear, the metal matrix is reinforced with the additional material. It’s referred to as a hybrid composite when there

are three or more constituent parts present. A lighter metal, such as magnesium, titanium, or aluminum, often serves as the matrix in structural applications. High temperature applications commonly employ cobalt and cobalt-nickel alloys. Typically, an MMC divides manufacturing into three categories: liquid, vapor, and solid. Continuous carbon, silicon carbide, or ceramic fibers are a few materials that can be placed in a metallic matrix material. MMCs can function in a broad variety of temperatures, have greater electrical and thermal conductivity, and are fire resistant. Applications have also been shown to be radiation and out gassing-resistant. Most metals and alloys provide suitable matrices for composite applications. There are three subtypes of polymer matrix composites (PMCs): rubber, thermoset, and thermoplastic. A large molecule known as a polymer is formed of covalent chemical connections joining repetitive structural units. A dispersed phase of fiber reinforcement and a polymer matrix combine to form PMCs. They can be produced more cheaply and easily. PMCs are less dense, more resistant to air and other forms of corrosion, and more effective at preventing the conduction of electrical current than metals or ceramics [Yang et al. 2012].

Composites can be of different types according to reinforcements as well. Particulate reinforced composite can be made by large particles or smaller particles and are cheaper to make. When strength to weight ratio matters, fiber reinforced composite can be deemed very useful. Fiber reinforced composite can be made with stiff fiber in the low-density matrix. Reinforcing fibers can be short or long. They can be oriented randomly or unidirectional or oven or of preferred orientation. Composite can also be made by making structure like laminar or sandwich. A laminar composite with greater isotropic strength in the plane is created by stacking and adhering sheets (or panels) with various orientations of high strength directions. Modern skis and plywood are two examples. Sandwich panel composites can be made with strong, stiff end sheets attaching to a light core structure, like honeycomb, which gives it shear strength. It is used in the construction of walls, roofs, and aircraft [Yang et al. 2012].

In recent world polymer matrix composite (PMC) is omnipresent. They are two types. Thermosets, thermoplastics [Sing et al.2020]. Thermoplastics, also known as thermos softening plastics, are polymers that become malleable or moldable above a certain temperature and return to a solid state upon cooling. Examples of thermoplastics

include low density polyethylene (LDPE), high density polyethylene (HDPE), polypropylene, nylon, and acrylics. Thermoset plastics are synthetic materials that can be heated to a point where they get stronger, but once that point is reached, they can no longer be properly remolded or heated again. On the other hand, thermoplastics work in the exact opposite way, softening when heated and hardening and strengthening when cooled. Repeated heating, shaping, and cooling of thermoplastics does not alter their chemical composition, however heating thermosetting polymers after the first molding will cause them to burn [Kessler 2012].

As the increase of PMC is increasing day by day it is creating wastes that is not biodegradable and the process of destroying these PMC is more harmful to the nature as they release a lot of harmful chemicals to the environment [Chakraborty et al. 2022]. To reduce the non-biodegradable nature, a more environment friendly way is to use natural fiber in the polymer matrix. Usage of natural fiber reduces the carbon footprint and greenhouse gas emission [Gholampour and Ozbakkaloglu 2020]. Composites made of natural fibers offer an alternative to synthetic materials that are bad for the environment and help reduce pollution. Aside from that, using Natural fiber reinforced polymer composite (NFRPC) in construction can reduce construction wastes [Sanjoy et al. 2018].

Ramnath et al. [2014], Sanjay et al. [2015, 2016], Yelin et al. [2016], Neto et al. [2022] reported that the natural fiber composites exhibit improved electrical resistance, good mechanical properties, good thermal and acoustic insulating properties, as well as higher resistance to fracture. Despite these benefits, there is a significant disadvantage to natural fiber. They have low dimensional stability and are particularly sensitive to moisture. Plants produce lignocellulosic natural fibers, which are composed of cellulose, hemicelluloses, lignin, pectin, and waxy substances. The primary structural component of the fiber structure is thought to be cellulose. Strength, stiffness, and structural stability are imparted to the fiber [Kabir et al. 2012].

Natural fibers derived from plants are lignocellulosic in origin and made of cellulose, hemicelluloses, lignin, pectin, and waxy materials. The three hydroxyl groups (OH) that make up the chemical structure of cellulose are present. Two of them establish intramolecular hydrogen bonds with the cellulose macromolecules, and the remaining members of the group create intermolecular hydrogen bonds with other cellulose

molecules. Hemicellulose comprises branching polymers with five and six carbon sugars) and a variety of chemical configurations, and it is mostly found in the major cell wall.

The lignin's aromatic structure is amorphous. Polysaccharides with complex structures make up pectin. Their side chains are cross-linked by the calcium ions and arabinose sugars. Additionally, traces of organic (extractives) and inorganic (ash) materials are present in the fiber structure. While organic extractives are in charge of the fiber's colour, fragrance, and resistance to decay, inorganic components increase the fiber's abrasiveness. According to the kind of fiber, different cellulose microfibrils have different cell shapes, which is what gives the fiber its distinctive properties [Kabir et al. 2012].

Cellulose microfibrils are found in main and secondary levels in each fiber cell wall. The main cell wall is where the fiber structure grows and is deposited. Each of the three layers that make up the secondary wall has a lengthy chain of helical cellulose microfibrils. Gradually from main to secondary layers, with equivalent amounts of hemicellulose in each layer. However, in this order, the lignin content drops. Hemicellulose molecules produce cementing elements for the fiber structure when they hydrogen bind with cellulose fibrils. The cellulose-hemicellulose network is connected with lignin and pectin, which provide the molecules an adhesive characteristic to hold them together. The strength and stiffness of the fiber are a result of this adhesive property. The mechanical characteristics of the fiber are determined by the secondary thick layer. In general, fibers with more cellulose and a smaller microfibrillar angle (angle between the fiber axis and the cellulose microfibrils) have stronger physical characteristics [Kabir et al.2012].

Crystalline and amorphous areas help to differentiate the cellulose structure of the fibers. In the crystallite area, many strong intramolecular hydrogen connections are created. As a result, cellulose blocks are formed, making it challenging for other chemicals to penetrate. The amorphous area, however, readily absorbs colours and resins. The water molecules from the atmosphere are mixed with the hydrophilic hydroxyl groups that are present in this area. These water molecules are often held by hemicellulose, lignin, pectin, and waxy materials. This changes the fiber's nature to make it hydrophilic and polar, thereby decreasing its compatibility with the non-polar/hydrophobic matrix. Natural fiber has to be chemically treated for the expansion of the crystalline area, the removal of the

hydrophilic hydroxyl groups, and the removal of surface contaminants (waxy substances). The surface and structure of fibers are frequently modified using chemical processes such mercerization, acetylation, benzoylation, peroxide, and coupling agents with or without heat. At roughly 240^{o}C, natural fiber often begins to break down. The structural components of fiber (cellulose, hemicelluloses, lignin, etc.) are sensitive to a variety of temperature ranges. Hemicelluloses and other cellulosic materials deteriorate at temperatures above 200^{o}C, according to a report. Lignin begins to break down at lower temperatures. By eliminating a particular percentage of the hemicelluloses and lignin components using various chemical processes, the fiber's thermal stability can be improved [Kabir et al. 2012].

The length of the fibers, the amount of stress, their orientation within the matrix and the way they are chemically treated are some of the variables that affect the mechanical characteristics of composites. Stress is transferred through shearing at the interface along the length and ends of the fibers when a load is applied to the matrix. The crucial fiber length (aspect ratio), the direction and orientation of the fiber, and the compatibility of the fiber-matrix contacts all affect how much weight is transferred. Three different types of composites are created depending on the orientation of the fiber at the matrix. First off, since the fibers buckle more easily, longitudinally oriented fiber composites often have better tensile strength but lower compressive strength. Second, the tensile strength of transversely oriented fibers is much lower than the strength of the matrix. Last but not least, short fiber composites with random orientations exhibit various mechanical characteristics. This is because it is much harder to maintain the mechanical characteristics of these composites due to the complexity of load distribution at the surfaces. Considerable changes in characteristics may be made by regulating elements like the dispersion, aspect ratio and orientation of fibers **[**Mwaikambo and Ansell 1999, Joseph et al. 2003, Fakirov and Bhattacharyya 2007]

Air or other volatile chemicals may become trapped inside the composites during the insertion of the fiber into the matrix. Following the curing process, micro-voids develop along the individual fiber tows and in the areas with a high concentration of matrix. The composites breakdown suddenly as a result, and their mechanical qualities are subpar. The creation of voids in composite materials is partly a result of their curing and

cooling rates. Low fatigue resistance, affinity for water diffusion, and increased variance (scatter) in mechanical characteristics are all caused by high void content (above 20% by volume). Composites with a larger percentage of fiber run the risk of developing voids greater [Amjad et al.2022].

Processing methods for natural fiber composites are, in theory, comparable to those used for processing synthetic fibers. Randomly oriented (short), unidirectional (raw and carded), and woven textiles are employed as reinforcements in thermoset and thermoplastic matrices, primarily on the length, orientation, and kind of the fiber. “Hand layup” is the standard manufacturing technique for thermoset composites. This hand mixing of the matrix and fiber takes place. The level of craftsmanship used in this process determines how consistent the composite is in terms of thickness, the ratio of fiber to matrix, and the number of voids present throughout the sample. As an alternative, resin is drawn inside under vacuum pressure and combined with the fibers mats in vacuum assisted resin transfer molding (VARTM). Under these circumstances, a composite’s resin impregnation quality is far superior than that produced by hand layup, and the void content may be reduced to a minimum. With this technique, the composite’s dimensions and fiber spacing are accurate. For both thermosets and thermoplastics, pultrusion is a different process. The reinforcement is forced through a heated die while being combined with the matrix to create the composite profile. Fiber-resin is added as granulate to the machine, melted into a fluid mass, and then injected under high pressure into the form. These processes are used for compression molding and injection molding, respectively, for the thermoplastic matrix. These procedures required pressure and high temperatures (over 200^{o} C) to fully bind the fiber and matrix [Adekunle et al. 2010].

When two phases are linked mechanically or chemically, a zone of diffusion or reaction occurs at the fiber-matrix interface. Interfacial adhesion essentially defines the mechanical properties of the composites. Low adhesion across the phase boundary results in weak force dispersion, which causes low mechanical properties. Many problems with the reinforcing of natural fiber into the matrix come as a result of the hydrophilic hydroxyl groups present at the interface. This hydrophilic nature interferes with the matrix’s ability to respond effectively [Wang et al. 2007]. Additionally, the reactive functional groups of the fiber are covered by pectin and waxy substances, which act as a barrier to the fiber's

ability to interlock with the matrix. The surface of the fiber must be altered with various chemical processes, reactive additives, and binding agents in order to improve interfacial bonding. Chemical processes reveal more reactive groups on the surface of the fiber, which promotes effective bonding with the matrix. Better mechanical characteristics of the composites can be obtained as a consequence [Das et al. 2000].

Alkali treatments are one of the most popular chemical treatment available for natural fiber reinforced composite. Plant fiber, also known as natural fiber, is mostly composed of cellulose, hemicellulose, lignin, pectin, waxes, and a few water-soluble substances. Despite their benefits, natural fibers have significant drawbacks that make them unsuitable for use in various composite applications. These drawbacks include poor compatibility, poor wettability, excessive moisture absorption, and heat deterioration during fabrication. Due to the low wetting between the hydrophobic organic polymer matrix and the hydrophilic natural fibers, one of the key downsides is the weak bonding at the contact of the reinforcement and matrix. Before creating the composite, the natural fiber surface needs to be chemically treated in order to solve this issue. One of the most popular chemical processes used to change and clean the surface of natural fiber to improve interfacial bonding and lower surface tension is mercerization or alkali treatment [Narayana and Rao 2021].

Using various mathematical and numerical methodologies, researchers are currently attempting to anticipate the natural fiber reinforced polymer composite's mechanical performance. Instead of expensive and laborious mechanical testing of composite materials, analytical and statistical modeling techniques are used to project and comprehend the behavior of the composite as a function of the characteristics of its components. Finding the ideal parameter becomes more time-consuming and unproductive, especially when considering the interactions between each component. Response surface methodology (RSM) can be used to address this problem. According to RSM has the capacity to define the effect of independent factors on the replies either by each variable alone or in combination throughout the process, as well as to assess the link between the responses and the independent variables. As compared to the conventional technique, utilizing RSM can also minimize the number of scheduled trials. It can be used to predict output response as well as optimize the input parameters [Aimi et al. 2014].

Chapter-2

Literature Review

2.1 Effect of Chemical Treatment on Fiber

Due to low cost and wide availability, interest in using natural fibers as fillers in composite materials is increasing quickly. However, Natural fibers can only be used in a limited number of applications due to weak surface adherence and mineralization. Therefore, it is crucial to carry out a treatment that can enhance the surface qualities of natural fibers before they are employed in composites. These therapies include physical ones (corona, plasma, etc.), chemical ones (alkaline, silane, acetylation, etc.) and others [Koohestani et al. 2019].

According to Xie et al. [2010], Choy et al. [2015], Koohestani et al. [2018], silane is an inorganic compound with the general chemical formula SiH4 that is made up of hydrophilic and hydrophobic compounds joined to the silicon molecule. Silane can be coagulated by adding a coagulant agent to create silanol (SiOH) groups at first and then siloxane bridges. Kabir et al. [2012] indicated that throughout the course of the treatment, it experiences a number of steps of bond formation, condensation, and hydrolysis. During the process, the hydroxyl group of the fiber is attacked by one end of the silanol, and the polymeric matrix may also be attacked by the organosilane's organic portion. Valadez-Gonzalez et al. [1999] found that Methacryloxy silane (A-174 from Sigma-Aldrich) treatment of henequen fiber at 70 °C increased mechanical strength. A 70% increase in tensile strength and a reduction in the curing time of polymeric composites were seen when bamboo fibers were treated with organosulfur silane (Si-69 from HB Chemicals). Additionally, a 13% increase in flexural strength of the henequen-HDPE composite was produced while the flexural modulus remained same [John and Anandjiwala 2008]. Investigation by Huda et al. [2008] shows that. The study of Atiqah et al. [2021] states that sugar palm fibers are subjected to a 3-hour treatment with 2% saline and 6% sodium

hydroxide. It suggests that following the treatments, the boundary contact between the fiber and thermoplastic polyurethane has improved. According to the research that were evaluated, the amount of silane agent employed varied between 1% and 5%, and the primary variables that determined how well the silane treatment worked were hydrolysis time, silane chemistry, temperature, and pH.

Another chemical treatment is acetylation treatment [Kabir et al. 2013]. The acetylation treatment works by replacing the fiber's hydroxyl groups with an acetyl group (CH_3CO), which eliminates moisture and creates hydrophobicity. Additionally, Pickering et al. [2016] found that acetylation treatment makes the fiber surface rougher to increase bonding between fiber and matrix. Ali et al. [2018] depicted that acetic acid need to be pre-treated by soaking before being treated with acetic anhydride because they are not reactive with natural fibers adequately. Higher levels of acetylation reduced the ability of flax fibers to absorb moisture, but 18% level of acetylation gave flax fibers the greatest tensile and flexural qualities [Bledzki et al. 2008].

Zaman and Khan [2021] revealed that the elimination of lignin and extractible, a tiny rise in the percentage of cellulose, and a small amount of hemicellulose converting to acetylated hemicellulose might all be contributing factors to the increase in tensile strength when the treated Banana fiber with Acetylation. Additionally, it was observed that the acetylation of Banana fiber eliminated waxy material from the outermost layer of the fiber, enhanced the strength of the interface between the fiber and matrix, and increased surface free energy, all of which were advantageous for better composite qualities. A study of Nair et al. [2001] shows that a one-hour experiment using preserved natural sisal fiber with 18% sodium hydroxide solution, glacial acetic acid, and two droplets of concentrated H2SO4 in acetic anhydride. The sisal fiber's treated surface appeared to become heavier and had many gaps that helped it mechanically adhere to the polystyrene matrix. It has been suggested to create a possible prototype of the sisal fiber/polystyrene compound interface.

Tserki et al. [2005] suggested, acetylation treatment of flax and hemp fibers reduced their moisture absorption, eliminated the fibers’ non-crystalline components, produced a smooth fiber surface, and increased the efficiency of stress transmission. Additionally, according to the research done by Zafeiropoulos et al. [2007], acetylation altered both the surface and bulk characteristics of flax fibers. Zaman and Khan [2021]

found that alkali and acetylation treated banana bunch fiber polypropylene composite got more thermal stability than normal polypropylene. Study of Joseph et al. [2005, 2006] showed that the composite's tensile strength rose when the banana fibers were acetylated, but not the individual fibers' tensile strength. Additionally, the treatment also caused an increase of fiber mass up to 16.3%.

One of the most commonly used chemical treatment is alkaline treatment or mercerization [Adams et al. 2016]. In this process, sodium hydroxide (NaOH) is used to change the natural fibers' cellulose molecular structure [Edeerozey et al. 2007, Gholampour and Ozbakkaloglu 2020] The natural fiber's hydrogen bonding is broken up by this process, which also gives the matrix material a rough surface to touch. Better chemical connection between the two is brought about by the roughness. This cleans the fiber's outside surface of excess oil, wax, and lignin. In this process, sodium hydroxide (NaOH) is used to change the natural fibers' cellulose molecular structure [Jahan and Sony 2021]. It was mentioned in the work of Zalinawati et al. [2020] that alkali treatment changes the fibers composition and also responds to its structure. It entails submerging fibers in various concentrations of aqueous sodium hydroxide (NaOH), according to Setswalo et al. [2017] OH group of fibers reacting with NaOH, makes water, releases impurities and coats treated fibers with Na^+. Because the fibers and polymers adhere well to one another, there have been reports of improvements in the mechanical characteristics and fiber quality. The hydrogen bonds in the network structure are broken and the surface roughness of fibers increases as a result of alkalization [Sadhu and Gupta 2019]. It also alters the cell wall's microfibril consolidation, which strengthens the connection within the matrix and fiber [Vinod et al. 2020].

The elimination of waxes and natural lipids from cellulose fiber surfaces results in an increase in the amount of reactive functional groups [Salam et al. 2012]. It was also found by Jain et al. [2018] that after soaking Agave fiber for around 2 hours under temperature 23 °C with 8% concentration of alkaline solution, lignin, wax and water-soluble compounds were removed. To enhance the natural fibers' microstructural qualities, fiber treatment can be thought of as an accelerated aging process carried out under controlled circumstances. During the soaking cycle, cellulose swells, improving its dimensional stability. Additionally, the reduction in water retake inside the inter-fibrillar

area caused by the creation of an irreversible hydrogen bond with each cycle of treatment [Ballesteros et al. 2017]. Kallakas et al. [2015] also agreed that swelling causes fiber deconvolution, which affects the crystallites' orientation and helps relieve the internal tensions that are present in fibers as well as enhances chemical reactivity and adds shine.

In the study of Oushabi et al. [2017] Date palm fibers were subjected to treatment with sodium hydroxide (NaOH) at several concentrations; untreated, alkali 2%,5%, and 10%, respectively. The treatment took place at 25^{o}C for 1 hour, following which the fibers were repeatedly washed with distilled water to remove any remaining NaOH, and then dried in an oven at 80^{o}C for 1 hour. From the SEM images it was evident that 2% alkali treatment was not fully able to remove the impurities from the fibers, where as 5% alkali treated fiber was completely free of wax, oil and other impurities without harming the fiber surface as well as making the surface rougher to accommodate better bonding between fiber and matrix. However, after treating fibers with a 10-weight percent NaOH solution, it was discovered that the high alkali concentration had damaged the surface topography of the fibers. Study of Kai et al. [2015] also reach to similar calculation where Abaca fibers were treated with 5%, 10% and 15% concentration of alkali solution. The tensile strength start showing nonlinear behavior for the 10% and 15% alkali treated fibers because of reduction of diameter of fibers, which makes the fibers softer and reducing young's modulus but increasing the elongation for higher concentration of treated fibers.

Mwaikambo and Ansell [2000] found that after treating hemp, jute, sisal and kapok fiber with alkali, surface topology was improved with clean and rough surface in case of jute, sisal and hemp but no effect on kapok fiber. In the study of Fang et al. [2023] bamboo fibers were treated with different alkali concentration also varying soaking time. Alkali treatment concentration was one of the parameters examined that significantly affected the crystallinity and thermal stability of bamboo fibers. Temperature had a significant impact on bamboo fiber's crystallinity, but not on its thermal stability. Surface roughness was significantly influenced by every treatment element, although soaking time had the most effects. But it did not have significant effect for the crystallinity and thermal stability. When the NaOH concentration was less than 5 weight percent, it had no impact on the surface roughness. However, when the concentration was beyond 5 weight percent, the surface roughness increased even more.

Another study of Chen et al. [2018] depicted that individual bamboo fibers treated using low concentrations of NaOH might have improved moisture absorption, but bamboo fibers infused with high concentrations of NaOH (25%) had decreased moisture absorption. Thermal analysis showed that after the application of alkali at low NaOH concentrations (6, 8, and 10%), the beginning and peak of disintegration were moved to higher temperatures, indicating an improvement in the thermal stability of treated individual bamboo fibers, whereas the thermal stability had been compromised after alkali treatment at greater concentrations (15 and 25%). Investigation by Sunny et al. [2020] finds 51% and 62% increase of Tensile strength and Young's modulus of hemp fibers respectively, after high temperature treatment at 120 °C with 5 % NaOH and comparison to untreated fiber. The removal of more non-strengthening components from the fibers during the high temperature treatment than during the ambient temperature treatment is supported by SEM, XRD, FTIR, TGA, and change of diameter investigations. Better packing of the cellulose chains occurs for high temperature treatment, providing better resistance to cellulose degradation, as evidenced by the improvement of fiber strength with high temperature alkali treatment compared to the reduction of fiber strength obtained with ambient temperature alkali treatment and the increase in crystallinity index for fiber after high temperature treatment.

Study of Kim et al. [2012] found that, pretreatment with 1% NaOH for about 4 hours at 75^{o}C proved effective in removing lignin and other non-strengthening components. Devnani and Sinha [2019] conducted SEM, FTIR AFM, XRD, TGA, and mechanical property tests, 5% alkali treatment resulted in the greatest improvement in mechanical, morphological, and thermal characteristics for kans grass fiber. In another experiment tridax procumbens fibers (TPFs) were examined for their physical, chemical, crystallinity, thermal, wettability, and surface properties in both their raw and alkali-treated forms. According to the test findings, alkali treatment increased the amount of cellulose while decreasing the amounts of hemicellulose, lignin, and wax. This improved the qualities of tensile strength, thermal stability, crystallinity, and surface roughness. For treated TPFs, the contact angle was similarly smaller, demonstrating enhanced wettability with the liquid phase [Vijay et al. 2019]. In another chemical study of fibers extracted from the aerial roots of banyan tree (ARBF), cellulose concentration increased following alkalization whereas hemicellulose, lignin, and wax content decreased. The X-ray

diffraction examination showed that alkali treated ARBFs had improved crystalline size (7.74 nm from 6.28 nm) and crystallinity index (76.35% from 72.47%). The findings of the thermal study proved that the alkali-treated ARBFs' maximum degradation temperature (368 °C) and kinetic activation energy (75.45 kJ/mol) had increased from 358 °C and 72.65 kJ/mol, respectively. Following the alkali treatment, contaminants and wax on the outermost layer of the ARBFs were eliminated, according to the outcomes of scanning electron and atomic force microscopy [Ganapathy et al. 2019].

Loganathan et al. [2020] showed that NaOH-treated Cytostachysrenda fiber for soaking times of 1 and 2 hours, the NaOH concentrations have been determined as 1, 3, and 5 wt.%. Density, composition of chemicals, X-ray diffractometry (XRD), fourier transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA) were used to analyze the treated fibers. The findings showed that longer soaking times and higher concentrations increased density. The amount of amorphous hemicellulose and lignin has decreased after NaOH treatment. On the other hand, fiber treated at 3% NaOH for 1 hour showed an enhanced crystallinity index of 6% coupled with an increase in cellulose content of up to 0.27%. Study of Borchani et al. [2015] did a comparative analysis of untreated and 1% and 5% NaOH treated alfa stem, which revealed that the removal of various non-crystalline plant components caused some thermal, structural, and morphological changes in the fibers. After alkalization, surfaces appeared rougher in SEM. A progressive improvement in the cellulose level was seen by FTIR after alkali treatment with rising NaOH concentration. Additionally, it was discovered that treated Alfa fibers had better thermal stability and crystallinity index after being alkali treated. Another study of Saharan aloe vera cactus leaves conducted by Balaji and Nagrajan [2017] Shows that 5% alkali treated fiber shows better tensile result and amorphous materials were eliminated from according to FTIR spectroscopic examination. The alkali treatment improved the fibers' crystallinity Index and crystalline size, according to the findings of an XRD study. TGA was used to examine the fibers' thermal behavior. Following the alkali treatment of the fibers, the temperature for thermal stability and thermal degradation rises. SEM analysis of the fiber morphologies shows that alkali treatment causes the fiber surfaces to become rough. Study of Madhu et al. [2020] of chemically treated Agave americana fiber also shows increase in tensile strength and amorphous content reduction from FTIR analysis and shows better thermal properties from TGA analysis. The contact angle study

demonstrates how chemical treatment increases surface roughness, which leads to improved moisture retention in the treated fibers. In a Study of grape cane fiber, it shows that treating grape cane fiber with more than 3% weight of NaOH caused negative effects on the fiber's overall characteristics. Therefore, for fiber shape, cellulose content, crystallinity, and single fiber bundle tensile test, lower concentrations of NaOH (1 % for outer bark fibers and 3% concentration for inner bark fibers) generated superior attributes. [Bakar and Kamke 2020].

A different study of areca palm leaf stalk fiber shows the decrease of diameter and its density raised as the alkali percentage rose. According to the chemical characteristics, the amount of cellulose in each of the fibers rose as a result of an increase in alkali concentration, which also resulted in the removal of hemicellulose, lignin, wax, and other impurities. The partial elimination of hemicellulose, lignin, wax, and other contaminants from fiber surface was further supported by the FTIR investigation. The Areca palm leaf stalk fibers' tensile characteristics are enhanced by the alkali treatment. In comparison to untreated, 10%, and 15% NaOH treated, the 5%NaOH treated areca fiber had superior tensile characteristics. Tensile strength for the 5% alkali treated was 486.41 35.57 MPa, tensile modulus was 1.46 GPa, and elongation at break was 1.82% [Shanmugasundaram et al. 2018]. According to Narayanasamy et al. [2020], Calotropis gigantea fruit bunch (CGFB) fibers amorphous contents and non-cellulosic elements that were detected by the FTIR analysis were found to be reduced by the alkali-treatment. The crystallinity index of the alkali-treated CGFB fibers was relatively greater than that of raw fibers, according to the X-ray diffraction pattern. Alkali-treated Calotropis gigantea fruit bunch fibers have greater thermal stability and degradability than untreated fiber. Images obtained using scanning electron microscopy and atomic force microscopy revealed that the fiber's surface had been somewhat roughened as a result of the alkali treatment's eradication of surface contaminants and non-cellulosic materials.

A study conducted by Ilangovan et al. [2020] about sausage plant fiber depicts that use of NaOH remove the fruit's natural cellulose fibers, which were then bleached and examined for their characteristics. The alkali treatment and bleaching produced a highly cellulosic fiber (up to 71%), the results showed that a considerable quantity of hemicellulose and lignin was lost. Depending on the level of treatment, the surface of the

fibers changed morphologically from rough to smooth. Following the treatment, the thermal stability, crystallinity, and hydrophobicity all enhanced. In another study of Ebissaet al. [2022]. Y.alpina Ethiopia indigenous bamboo species single fiber were treated with different concentration of NaOH and the best mechanical properties were achieved at 10% concentration by soaking 2 days, whereas all the concentration clearly shown reduction of lignin and hemicellulose contents. It was also seen that among the Y. alphina bamboo fibers that have been treated with 6–18% NaOH, the 12% NaOH concentration had highest thermal stability characteristics. Alkali treatment changed the cell wall's microfibril formations and surface appearance. The alkali treatment's effects on the composition, structure, and characteristics of sugarcane bagasse fibers were demonstrated by the results. lignin and hemicellulose content drop, while the number of cellulose increases to its highest point in relation to the alkali content of the treatment solution.

Contrary to numerous findings in the literature, crystallinity only little alters and microfibril angle stays constant as NaOH concentration rises. The strength of the fibers reaches a significantly more noticeable maximum at around 5-8 wt.% NaOH concentration, whereas stiffness reaches a very small maximum at about 2-4 wt.%. The fiber strength has increased significantly. The breakdown of vulnerable amorphous fractions and the rise in cellulose content were used to explain the improvement in fiber characteristics. Around 5% weight percent is the therapeutic solution's ideal concentration [Bartos et al. 2020]. A study of Borassus flabellifer leaf fiber with different alkali treatment also found the change in functional group with the help of FTIR, rougher surface and porosity in fiber from SEM images [Abubakar et al. 2023].

2.2 Natural Fiber Reinforced Polymer Composite

For the past two decades the world is showing more concern about the pollution of environment and plastic material which do not degrade quickly, is creating a substantial problem. Synthetic fibers and just plain plastics are not that bio degradable and to solve that problem and reduce some of the cost of those synthetic fibers, researchers are showing their keen interest in natural fiber reinforced composites to consider the wight to strength ratio and biodegradability as well as their availability and low cost. Jute is one of the most popular natural fibers in the world and have been used in a lot of polymer composites as

well. Memon and Nakia [2013] by utilizing compression molding, jute spun yarn/Polylactic acid composites were created in a single direction. The matrix resin fiber and reinforcing fibers were paralleled and employed as intermediary materials to construct the unidirectional composite. The mechanical characteristics of the jute spun yarn/PLA composite were described, and cross-sectional observations were used to assess the fabrication performance. By raising the molding temperature and achieving a higher ratio of elastic modulus rise, penetration quality and fiber stack dispersion were improved. Due to the degradation of the jute fiber, the tensile strength achievement ratio fell when the molding temperature was raised. For the fabrication of the jute spun yarn/PLA unidirectional composite, the ideal compression molding temperature ranged from 185^{o}C to 195^{o}C.Using a needle-punched method, jute mat was put on glass mat to create the hybrid jute/glass mat. The layers of jute and glass mat were arranged in the following ratios: 1:0, 1:1, 1:2, and 2:1. After being needle-punched, a polyester resin that was unsaturated was used to create the composites using the hand lay-up technique. In order to determine the flexural characteristics of various composites, a three-point bending test was conducted. The findings show that the flexural strength of the jute mat composite (JF) was about 37 MPa, whereas the flexural strength of the jute mat/glass mat hybrid composites (JF/GF and JF/JF/GF, when the glass fiber layer is placed as bottom side) was much higher, showing values of about 61.7% and 62.0% higher than those of JF [Zhao et al. 2017]. The subject of study of Arao et al. [2015] were the mechanical characteristics of green composites made of jute fibers and polylactic acid (PLA). Short fiber particle composites, which have a substantial compound intensity, performed optimally mechanically.

Stalin and Shobhanadevi [2021] reported that jute fiber served as reinforcement, while cardanol resin served as the matrix, in the creation of a bio-composite. Through the use of scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA), the microscopic makeup and surface properties of the bio composites are studied. At 30% particle loading, TGA measurements showed that treated jute bio-composites had improved thermal stability (352°C and 361°C) in comparison to untreated jute bio-composites (447°C and 458°C). A hybrid composite was fabricated reinforced by sisal/jute/glass fiber using compression molding the interfacial shear strength, tensile strength, flexural strength, impact strength, and water

absorption tests were applied to describe these composites. By employing scanning electron microscopy analysis, microscopic examinations were conducted to examine the interfacial interaction between the fiber and the matrix, fiber dispersion into the matrix, and structure of the cracked surfaces. According to the findings, sisal and jute fiber hybridization with glass fiber can increase the qualities compared to sisal or jute fiber reinforcement with glass fiber in a single fiber. These composites' reasonable adhesion and physical qualities suggested that they might work well in the building, vehicle, and aviation sectors [Ramesh et al. 2016]. In a different study a thermosetting polymer (epoxy) was used as the binder material to create several specimens of jute- and chicken feather fiber-reinforced polymeric hybrid composites. According to ASTM standards, several physical and mechanical qualities of composite specimens were examined. Using SEM images, the morphology of their surface fractures was examined. The findings show that the polymer hybrid composite with 10 weight percent jute, 20 weight percent chicken feather fiber, and 70 weight percent epoxy matrix had a maximum closeness factor of 0.661048, indicating a high interfacial strength and binding between the fibers and matrix, leading to favorable overall mechanical tensile, compressive, and impact hardness characteristics density and moisture absorption [Johri et al. 2019].

Demonstration of Ali et al. [2019] about experimental and computational modeling of the flexural behavior of composites made of carbon and jute shows that flexural strength falls as jute content rises. More than 10% of the experimental data differ from the simulation of flexural behavior. This anomaly is brought about by the waviness of the fiber, which causes a heterogeneous distribution of properties in composites. Khalid et al. [2020] reported that carbon fiber composites can be replaced with jute fiber hybrids without suffering a major reduction in tensile strength. The nondestructive material characterization method of simulation offers high potential for tensile response prediction. Through destructive lab testing, 90% of the material costs needed for tensile characterization may be avoided. Due to the existence of voids and the brittle nature of epoxy, SEM research showed that the matrix breaks in the form of fragmentation, with fibers pulling out after failure. Study of Sathees [2020] of sorghum bicolor, sisal fiber, and jute reinforced with polyester composites are detailed for the first time in terms of their tensile, flexural, impact, and hardness properties. The hand lay-up method is used to create the hybrid composite plates for various fiber weights. Tensile, impact, flexural, and

hardness tests were carried out in accordance with ASTM standards to study the mechanical characteristics. The findings of the mechanical tests showed a consistent pattern of increasing tensile, flexural, impact, and hardness qualities with the addition of natural fibers. Excellent resistance capability is also due to the natural fibers' strong adherence to the polyester matrix. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) have also shown the composite material's thermal stability and thermal disintegration. The work of Subramanian and Sekaran [2022] provides examples of mechanical, thermal, and dynamic mechanical analysis of alkali-treated hybrid jute, hemp, and kenaf filaments constructed with polyester amalgam. Based on the testing findings, the composite specimens' tensile strength, flexural strength, hardness, and impact characteristics were gradually improved, achieving maximum values of 194 MPa, 98 MPa, 98, and 29 J/m^2 respectively.

The hand lay-up approach was used to effectively create a novel class of polyester-based composites filled with jute and glass fibers, which shows with an increase in fiber loading, the hybrid glass-jute polyester composites' density falls. Maximum tensile strength occurs at 0° fiber orientation and 40% fiber loading, according to analysis of the mechanical characteristics of composites. However, at 60° fiber orientations and 40% fiber loading, flexural strength is at its highest. SEM image depicts that the failure of composites under tensile loads is clearly caused by fiber failure, a poor fiber-matrix interface, and voids, whereas failure under bending loads is caused by cracking of the matrix and fiber-matrix interface de-bonding. This is evident from scanning electron microscopic analysis of the fracture surfaces of composites [Purohit et al. 2023]. Research was conducted with the mixture of jute and cardanol resin which shows the jute composites with a 30% weight and 5% alkali treated has higher thermal properties. This is brought on by the cardanol resin and jute fiber's increased interfacial adhesion and dispersion [Stalin et al. 2021]. A successfully fabricated jute-polypropylene composite treated with alkali with different fiber loading depicts the maximum mechanical properties with an increasing trend till 50% wt. But start deteriorating after that. The effect of fiber loading to increase the mechanical properties were evident here [Das et al. 2018]. Another very popular fiber that has been used for decades for making composite materials is coir fiber. A study of Kumar et al. [2021] of NaOH treated coconut coir fiber reinforced polypropylene composite showed improved load bearing capacity. Up to 10% fiber

loading, treated composites depicts superior tensile and flexural properties compared to pure polypropylene composite but in case of impact and hardness properties, they failed to show superiority.

In a study of hybrid epoxy composite of coir, bamboo and jute it was depicted that in comparison to separate reinforcements, the hybridization of multiple fibers with epoxy matrix produced improved mechanical and thermal characteristics. The findings of the thermogravimetric study show that the thermal stability of each of the nine types of hybrid composites is almost identical, and little differences in degradation temperatures were seen in the thermographs [Sathis et al. 2022]. In another study hybrid composite of coir and jute with epoxy shows that greater weight percentage of jute fiber in the composite with a composition of 85% jute and 15% coconut results in better tensile strength. In the composite of 50/50 jute and coconut fiber content, it exhibits semi-brittle and ductile behavior. As a result, it was determined from this investigation that composite specimens are inherently more durable than pure coir specimens [Singh et al. 2021]. In another study of tampico and coir fiber with NaOH treatment and different fiber loads with linear low-density polyethylene (LLDPE) matrix, composites were fabricated and it showed that with 5% fiber load produces the best tensile, flexural and impact properties compared to unreinforced, 10% and 15% fiber load composites. 15% fiber load composites also depict voids, moisture bubble and fiber–matrix de bonding [Abhilash and Singaravelu 2020]. In a different study, Coir fiber and Sugarcane leaf sheath (SLS) fiber with different proportion of coir and sheath fiber (25/75, 50/50 and 75/25) with 30% fiber load was mixed into polyester matrix and the analysis of the data shows that the 50/50 (Coir/SLS) hybrid composite has superior mechanical qualities over other combinations. There are indications that adding both fibers to the polyester resin matrix will provide the material better mechanical qualities than using only polyester resin [Arul et al. 2020].

Bamboo has garnered a lot of attention among natural fibers as a possible reinforcement for various polymer matrices due to its good mechanical characteristics [Mousavi et al. 2022]. In an experiment using bamboo fiber-reinforced polyester composite, it was discovered that the composite with 25% wt. of fiber load exhibits the highest levels of tensile strength, young modulus, flexural modulus, and impact strength, while the composite with 20% fiber load exhibits less pullout and de-bonding in

micrographic images [Santosh et al. 2022]. According to Supian et al. [2021]. The mechanical and physical qualities of hybrid composite materials have been improved by the addition of stronger, more rigid bamboo fiber. Furthermore, compared to date-palm fiber epoxy composites without hybridization, testing results of the date-palm/bamboo epoxy composite showed enhanced characteristics. In a different study of hybridization composites were made using three different kinds of olive fibers: bamboo fibers (B) and olive tree short branch (OTS), big branch (OTB), and leaves (OTL). In comparison to pure fiber composites, it was discovered that the thermal stability of epoxy composites enhanced with the introduction of hybrid fibers. At the highest decomposition temperature, hybrid composites (OTB-B and OTS-B) show weight losses of 54.65% and 54.53%, respectively, compared to hybrid composite (OTS-B), which showed a lower residue (15.82%) [Rashid et al. 2022]. In an experimental investigation involving treated and untreated bamboo fiber (BF) reinforced poly butylene succinate (PBS), Young's modulus rose as BF concentration increased, but elongation at break dropped. The bio composites' Izod impact strength was comparable to that of pure PBS. PBS/BF bio composites' ability to absorb water improved as BF component rose. Due to the removal of lignin and hemicellulose from the BF surface, alkali-treated BF absorbed more water than untreated BF [Pivsa-art and Pivsa –art 2021].

In the agricultural industry, rice is a significant food crop that is related to cereal grains like wheat, barley, and oats. It occupies around 1% of the planet's surface. It is also the staple food in many Asian countries. In a study using rice husk and polystyrene composites, it was shown that moisture absorption increased with time and that composites with more fillers absorbed more moisture than those with less fillers. Microstructural investigation showed that 40 weight percent of filer loading produced the optimal fiber-polymer combination [Ighalo et al. 2021]. In a different experiment of rice husk (RH) and coco peat (CP) reinforced acrylonitrile-butadiene-styrene (ABS) composite observed the addition of single and hybrid RH and CP reinforcements had a detrimental effect on the composites' crystallinity and thermal characteristics. In contrast to plain ABS, all reinforced composites were more amorphous, and the thermal stabilities decreased. As the RH and CP particles were absorbed into the matrix, the chemical and dynamic mechanical characteristics were noticeably improved because of emergence of novel chemical structure and intermolecular interactions shown in the FTIR spectra [Haris et al. 2022].

Another research evaluates the effects of ethylene-acrylic-ester-maleic anhydride (E-AE-MA) and PP-g-maleic anhydride (MAPP) on the mechanical characteristics of polypropylene (PP) composites filled with rice husk (RH) and rice husk ash (RHA). According to the findings, PP/RHA composite showed a 4% significant improvement in tensile strength compared to PP/RH composite, whereas PP-RH composite showed significant increases in flexural and impact strength of 5% and 3%, respectively [Ezenkwa et al. 2022]. Reports from Boonsuk et al. [2021] depicted that, the tensile strength of thermoplastic cassava starch (TPS) reinforced with alkali-treated rice husk (RH) 20% wt. was significantly increased, pointing to efficient matrix/filler load transfer that can be linked to better matrix-filler interface interactions. Additionally, this class of TPS/treated RH composites demonstrated improved water absorption and maintained the outstanding biodegradable performance of the virgin matrix. In another investigation of 2% NaOH treated rice straw epoxy composite with 30% fiber load showed better tensile strength, flexural strength, flexural modulus, and young's modulus than untreated and hot water treated epoxy rice straw composite [Ranjan et al. 2021].

Pineapple fiber is another common fiber available in south east Asia. In a study of pineapple leaf fiber (PALF) reinforced polyester composite different composites were manufactured varying fiber length. As a result of a change in chemical structure in PALF, SEM results showed that the application of silane treatment produced better surface topography (fiber lengths of 5–10 mm showed smooth surface, resulting in crack proliferation with low fracture toughness of 15–32 MPa; whereas a fiber length of 15-20 mm produced better fiber–matrix bonding, improving the fracture toughness from 42–55 MPa). Because of the fiber-matrix interfacial connection, the 20 mm length of PALF produced improved characteristics (flexural, tensile, impact, and wear resistance) [Anand et al. 2022]. By creating composites with epoxy containing 5 vol.% graphite and 30 vol.% PALF and 10 vol.% graphite and 30 vol.% PALF, the combined effects of graphite filler particles and pineapple leaf fibers (PALF) on the mechanical behavior of epoxy composites were examined in another study. In comparison to other composites, epoxy composites reinforced with 30% PALF and 10% expanded graphite filler displayed better tensile and flexural performances. Additionally, the ductility of epoxy with the PALF fibers and graphite fillers was somewhat reduced [Reddy et al. 2021]. As a result of hybridization, kenaf and pineapple leaf fiber reinforced polypropylene composite

demonstrated improved water absorption and mechanical capabilities in a research investigation [Feng et al. 2020]. Jagadish et al. [2018] revealed that short pineapple leaf fibers (between 1 and 2 mm) are utilized as reinforcement and epoxy resin (Lapox (L12) resin + K6 hardener) as the matrix material. Six distinct fiber compositions are as follows: 0, 1, 5, 10, 15, and 20 wt.%. The mechanical characteristics of each composite specimen, including tensile strength, flexural strength, toughness, and hardness values, are then assessed. The outcome demonstrates how adding short fibers may enhance mechanical capabilities, and it is evident that composites with 10% reinforcement + at 5-mm thickness perform better than other combinations in all circumstances. Another experiment with pineapple leaf fiber (PALF) reinforced polyester (PE) composite reveled that in accordance with the law of mixing, the tensile, compressive, and flexural characteristics significantly increased with increasing PALF loading. Additionally, SEM pictures of samples with tensile fractures reveal considerably reduced fiber pull-out, fiber breakage, and enhanced fiber/matrix adhesion as a result of efficient stress transmission with the higher PALF loading in PE composites [Senthilkumar et al. 2019].

Another widely distributed species in south east Asia that is primarily utilized to produce banana fruit is the banana tree. It has long served as a source of nutrients. Banana plants have an extremely brief lifespan and often only produce fruit once. Since they are chopped after just one harvest, a highly rich source of natural fibers that can strengthen different polymer matrixes is permanently lost. Researchers are looking into the usage of banana stem and leaf fibers, and it is becoming more and more common in the field of fiber-reinforced polymer composites. In a study of vacuum moulded untreated and treated banana fiber reinforced epoxy composite, the surface treatment increased the mechanical characteristics [Subramanya et al. 2020]. Another investigation using compression-molded banana fiber reinforced epoxy composites with varied fiber loads (5, 10, 15, and 20% wt.) found that the composite with 15% load had the greatest tensile, flexural, and impact strength as well as the greatest degree of thermal stability up to 220°C [Balaji et al. 2020]. A separate study found that banana fiber reinforced polypropylene composites made by injection moulding and treated with 5% sodium hydroxide had superior mechanical qualities than composites that had not been treated. For tensile, flexural modulus, and flexural strength, a rising trend was also seen due to a rise in fiber load, however the inverse was true for tensile strength [Komal et al. 2018]. The thermal stability of

composites made of banana fiber (BF) reinforced acrylonitrile butadiene styrene (ABS), high impact polystyrene (HIPS), and high-density polyethylene (HDPE) was found by Mahesh et al. [2020] to be identical to the matrix. For the mechanical properties, BF reinforced composites' elastic modulus and flexural strength were also found to be increased for all three matrixes with little variation [Motaleb et al. 2021]. Another study compared banana fiber reinforced polypropylene composites with and without coupling agents and found that the latter had improved mechanical and thermal stability as well as absorbed more moisture.

The primary source of sugar production in many south east Asian nations is sugarcane, which is a highly well-liked fruit. Following the extraction of sugarcane juice, bagasse fiber is produced from the sugarcane trash. Due of its accessibility, several researchers are interested in using this fiber as a reinforcing agent in polymer matrixes. Surface roughness was discovered to be enhanced in a research of sodium hydroxide, alkaline KMnO4, and phosphoric acid treated bagasse fiber reinforced epoxy composite. Compared to other treated and untreated composite, alkaline KMnO4 treated composite shown improved thermal and tensile performances [Vidyashri et al. 2019]. In a study of NaOH-treated bagasse fiber with polymers, it was found that increasing the duration of the alkali treatment decreased the fiber's cellulose content, which in turn increased the ultimate tensile stress of the composite. The study also found that increasing the alkali concentration also increased the composite's ultimate tensile stress [Vignesh et al. 2018]. Another study using fiber reinforced phenolic composites made from sugarcane bagasse (SCB) and oil palm empty fruit bunch (OPEFB) shown that the performance and characteristics of OPEFB/SCB fiber composites are superior to those of pure fiber composites [Ramlee et al. 2019]. In a separate study, coir and sugarcane leaf sheath (SLS) fiber reinforced polyester composite was created using variable amounts of coir and SLS (25/75, 50/50, and 75/25) while maintaining a 30% weight percentage for all the fibers, demonstrated the best tensile and impact strength for a 50/50 mixture [Arul et al. 2020]. Another study found that the tensile and flexural strength of a hybrid composite made of polyester matrix and 10% bagasse fiber with increasing amounts of rice husk powder (0%, 3%, 5%, 10%) was inferior to pure resin because of void creation [Hemnath et al. 2021].

2.3 Areca Fiber Reinforced Polymer Composite

Areca husk or betel nut husk fiber looks to be a growing fiber among all natural fiber reinforcing materials since it is affordable, widely accessible, and has a very high potential perennial production. Areca Catechu Linnaeus is the scientific name for areca, which is a member of the palm family's Arecoideae subfamily. The weather and soil of South East Asian nations like India, Bangladesh, and Sri Lanka, among others, are excellent for growing area. The areca fruit and betel leaf are consumed by people all throughout these countries specially in Bangladesh, while the husk fiber—which is rich in natural fiber that may be utilized as reinforcement in polymer composite—is discarded as trash. Researchers are very interested in determining the potential of areca fibers in various composite manufacturing applications. Yusriah et al. [2012] investigated the impact of fiber maturity and its potential in diverse applications, evaluating the mechanical characteristics for husk fibers from fresh, ripe, and dried betel nuts. The chemical composition is reported alpha cellulose 53.20%, hemicellulose 32.98%, lignin 7.20%, fat and wax 0.64%, ash 1.05% and other materials 3.12% on average. the fiber length reduced as the maturity level rose, while the density increased from raw to ripe to dried fibers (5.7, 5.5, and 4.9 cm fiber length with densities of 0.19, 0.34, and 0.38 g/cm3, respectively). Raw fiber had a high-water uptake behavior because its larger lumen size supports the absorption of greater water content. Ripe fiber had a lower water uptake behavior because its lumen size is less than that of raw betel nut husk fiber, and dry fibers had the lowest water uptake behavior because of their compact size.

Not only areca husk but also areca sheath and leaf fibers are also used for reinforcement in polymer composite. Composites were created employing various fiber orientations in a study of areca sheath fiber reinforcing epoxy matrix. The composite with the sheath fiber orientation of 90°-90°-90° had the maximum strength and was calculated to have a Young's modulus of 1.798GPa. It also had the highest degree of elongation, with a value of roughly 11.6% relative to its initial length. Less fiber orientation in a composite causes it to have a lower longitudinal Young's modulus, which makes it more brittle and more susceptible to failure when under stress [Dinakaran et al. 2019]. In another study, a polyvinyl alcohol (PVA) composite reinforced with short areca sheath (AS) fiber treated with benzoyl chloride was created using different fiber loads. Using silica sand particles to

examine wear behavior, PVA/AS composites showed the most severe erosion rate at a 45° impact angle, regardless of fiber loading, showing semi-ductile behavior. When compared to other produced composites, composites with a fiber loading of 10 weight percent showed the least degradation [Nayak and Mohanty 2020]. In a different investigation, areca leaf sheath fiber reinforced epoxy resin was created using 30% fiber and 70% resin, and its tensile strength and young modulus were both 16.15 N/mm2 and 3345.16 N/mm2, respectively [Chethan et al. 2016]. Another study using areca leaf sheath fiber and epoxy resin found that the optimal load to achieve the best tensile, flexural, and modulus properties, as well as an increase in impact strength, was 50% (among 20%, 30%, 40%, and 50%). The other combination produced unsatisfactory results as a result of void formation, demonstrating the urgent. need for a void-free production environment [Anand et al. 2022]. Fiber content was found to be the fabrication parameter that had the greatest impact on both the tensile and flexural strength in a study of areca fine fiber reinforced phenol formaldehyde composites with varying fiber content, fiber length, alkali concentration, and alkali treatment duration using Taguchi's experimental design (L27 orthogonal array). The tensile and flexural strength of the composite are significantly influenced by the interplay between the manufacturing factors. The ideal set of fabrication parameters for achieving the highest tensile and flexural strength was discovered to be fiber length = 10 mm, fiber load = 35 vol%, alkali concentration = 6%, and treatment duration = 1 h [Mohan et al. 2017].

Study of Yusriah et al. [2015] reported, betel nut husk (BNH) fiber reinforced vinyl ester (VE) composites using different fiber orientation and fiber loads showed that the random short fiber ripe BNH reinforced VE composites produced the lowest thermal conductivity when compared to random nonwoven and unidirectional ripe BNH fiber-reinforced VE composites because the random direction of the fiber alignment in the random short fiber ripe BNH reinforced composites. Because the resin transition peaks for the BNH-reinforced VE composites were lower than those for plain VE, the addition of ripe BNH fiber to the VE resin decreased the heat stability of the VE composites. Compared to the impact of fiber content and fiber orientation, fiber maturity was shown to have a relatively minor impact on the flexural characteristics of the BNH-reinforced VE composites According to a different research betel nut glass fiber reinforced polyethylene composite was manufactured varying fiber loading (5%,10%,15% and 20%) and

evidenced that with fiber loading, the tensile strength declined while the Young's modulus rose. On the other hand, increasing fiber loading, flexural strength, flexural modulus, and hardness rose. The fiber-polyethylene matrix's morphological properties were revealed by SEM investigation. The composite that included the highest mechanical qualities (tensile, flexural, and hardness) was that which contained 20 weight percent of fiber at a 1:3 ratio of betel nut and glass [Haque and Hasan 2016]. According to studies using sisal, roselle, and areca fine (AFF) fibers as reinforcing agents in a phenol formaldehyde (PF) resin-based polymer matrix, the mechanical properties of the AFF/PF composite significantly improved when they were hybridized with sisal fibers [Athijayamani et al. 2015].

2.4 Cotton Fiber Reinforced Polymer Composite

The most frequently utilized and well-liked natural fiber and unchallenged ruler of the world's textile industry is cotton. But it's use in natural fiber reinforced composite manufacturing is still pale to that comparison. Researchers are still trying to find its full potential to apply in this sector. In a study of cotton and bamboo fiber reinforced epoxy composite made by compression molding with different fiber loading, in comparison to cotton/cotton composites, cotton/bamboo composites with a fiber loading of 45 wt. % showed greater fiber matrix addition and mechanical properties [Aruchamy et al. 2020]. Another investigation using three different NaOH concentrations and three different soaking times on cotton fiber reveals that the 1.5 M concentration with 3 h soaking time produces the highest tensile strength, which is around 53 MPa for 0° and 30 MPa for 90° directions. For a concentration of 1.5 M with a 1-hour soaking time, the young modulus values peak at 5120 MPa and 4240 MPa for 0° and 90° directions, respectively. The photos show that alkaline treatment enhances the interface by minimizing the air gaps between cotton fibers and epoxy resin. Fiber-matrix interactions are also identified by SEM examination [Baccouch et al. 2020].

In accordance with research of Alomayri et al. [2013] about geopolymer composites reinforced with cotton fibers, it was found that adding more fibers—specifically, 0.5% weight of fibers—increased the geopolymer's mechanical qualities while, on the other hand, reducing its density. According to Hashmi et al. [2007] cotton and graphite reinforced polyester composite depicted reduction of specific wear rate and

coefficient of friction. The wear resistance of polyester reinforced with variable weight percentages of graphite and constant cotton reinforcement was examined in a different investigation, and the results showed that adding graphite in this case, 5% wt. Increased the wear resistance[Parikh and Gohil 2017].Isophthalic polyester reinforced with cotton fibers manufactured by hand layup demonstrated the highest tensile and flexural capabilities for 25% weight of fiber load in an evaluation of caustic soda treated varied wt.% of fiber load [Sabinesh et al. 2014]. Another study showed how cotton composites might be filled with enzyme-treated hemp fiber (HF) microparticles and reduced graphene oxide (rGO) nanoparticles to enhance their mechanical characteristics. The cotton/epoxy composite with 3% weight percent of rGO exhibits an increase in tensile strength of 11%, flexural strength of 20%, and impact strength of 30%. The cotton/epoxy composite has improved tensile, flexural, and impact strength by 20%, 14%, and 116%, respectively, when loaded with 3 wt.% of HF particles [Kamble et al. 2021]. A separate investigation made it clear that 30% weight glass and cotton fiber reinforced epoxy composites had better mechanical qualities than 20% weight reinforced composites. It demonstrates how effective hybridization is [Giridharan and Jenarthanan 2019].

Balaji [2017] reported that among the coir-cotton-unsaturated polyester combinations, the cotton-unsaturated polyester composite had the highest tensile strength, measuring 30.97 MPa. The tensile strength of the composite material increased linearly as cotton fiber was added. Maximum flexural strength of 15.90 MPa was demonstrated by the cotton-unsaturated polyester composites, which was attained with a displacement of 1.50 mm and a force of 124 N. In composites with a high cotton percentage and a low coir fiber content, greater flexural strength was seen. The cotton-unsaturated polyester composite also achieved a maximum impact strength of 4 joules, and the impact strength was enhanced by adding more cotton fiber to the mixture of coir fiber, unsaturated polyester, and other fibers In a different study of 20 and 30% fiber wt.% woven cotton fiber (CF) reinforced polypropylene (PP) composite fabricated by compression molding by multiple layers of CF and PP sheets showed increased tensile strength from 21.87 to 28.07 MPa, tensile modulus from 618 to 1867 MPa, flexural strength from 21.7 to 45.3 MPa, and flexural modulus from 813 to 1925 MPa for 30%wt. However, the tensile elongation at break decreased marginally from 26.72% to 20.99% for the same [Rukmini et al. 2012]. Another different study of hemp-cotton reinforced epoxy composite with varying fiber

load depicted composites having 20 weight percent of fine hemp fiber with 15% cotton demonstrated enhanced peak tensile load, ultimate tensile strength, and percentage of elongation. The flexural load, flexural strength, flexural modulus, and impact energy properties of composites containing 30 weight percent of fine hemp fiber with 5% cotton were better to those of composites containing various weight percentages of fine hemp fiber [Madurai et al. 2021]. In order to improve the nonwoven cotton/poly (lactic acid) (PLA) fiber mats' electrical, thermal, and abrasion-resistance capabilities, it has been reported that they functionalized with graphene oxide nanosheets utilizing a simple dip-coating process and heat reduction. Manufacturing procedures for creating bio composites and adding functionality are easily adjustable. According to experimental findings, bio composites were significantly increased in abrasion resistance, electrical conductivity, thermal conductivity, and diffusivity when less than 0.5 wt.% of graphene nanoplatelets were added [He et al. 2022].

2.5 Modeling of Mechanical and Physical Properties

A potent statistical tool used in the planning and optimization of experiments is called response surface methodology (RSM). It offers a methodical way to investigate the connections between several input factors and an interesting response variable. RSM aids in comprehending a system's intricate relationships and nonlinear behavior. RSM provides prediction and optimization of the response variable within the experimental domain by fitting mathematical models to experimental data. This approach is used to optimize and enhance processes in a variety of industries, such as manufacturing, engineering, chemistry, and agriculture.

Through the use of response surface methodology (RSM), a technique built on the Box-Benhken design, the blending parameters of banana pseudo-stem epoxy composites were recently optimized. Using the ANOVA statistical method, the projected tensile strength value for these composites as a function of an independent variable was discovered. Fiber length, fiber content, and sodium hydroxide variables were shown to be substantially correlated in the 2 Factors Interaction (2FI) model, according to the findings analysis. The maximum tensile stress was calculated using this model, and the value of R^2 = 0.9973 showed that it closely matched experimental results. 3.25 mm fiber length, 5.45

(wt.%) sodium hydroxide concentration, and 29.86 (wt.%) fiber loading were shown to be the ideal parameters for tensile strength [Hassan et al. 2019]. In a different study for the purpose of forecasting and enhancing the variables affecting jute fiber reinforced concrete composite's (JFRCC) characteristics, a platform based on Response Surface Methodology (RSM) articulated with crow search algorithm (CSA) with standard-sized cubes and cylinders have been created by adjusting three independent variables, such as the amount of jute fiber, the duration of the water-cement mixture, and the water cement (W/C) ratio. The second order polynomial models for the compressive and tensile strengths of JFRCC were initially discovered using the RSM-based Box-Behnken Design (BBD). The ideal set of fiber length, fiber volume, and water-cement (W/C) ratio was determined to be 6 mm for fiber length, 0.2% for fiber volume, and 0.55 for W/C ratio. The anticipated maximal compressive and tensile strengths were found to be 35.1 N/mm^2 and 3.5 N/mm^2 (after 28 days of curing), respectively. Experimental verification of the expected ideal circumstances revealed that the experimental strengths utilizing the optimum values deviated by around 5% from the predicted values [Sultana et al. 2020]. In a different experiment to examine the mercerization impact of bamboo fiber-reinforced epoxy composites was designed using a Box-Behnken design (BBD) of the response surface methodology (RSM). Using analysis of variance (ANOVA) of the quadratic model, the projected tensile strength of bamboo fiber (Bambusa vulgaris) reinforced epoxy composite constructions was assessed. A significant regression for the coefficient between the variables was found after measuring a total of 17 experiment runs. Additionally, the treated and untreated bamboo fiber-reinforced epoxy composite triangular and square core constructions were put to the test under compressive pressure. It was discovered that, after soaking for 3.99 hours and drying for 72 hours, the ideal mercerization condition is 5.81 weight percent of NaOH [Hassan et al. 2020].

A study conducted by Avila et al. [2022] employing the response surface method (RSM)-based desire function technique for rice husk fiber-reinforced polymer composite (RFRC) drilling process optimization with four crucial input variables. According to the variance analysis (ANOVA), the experimental results were highly linked, with a 95% confidence level. In a different study the effects of fiber length and alkali dose on the displacement, flexural modulus, and flexural strength of plaster reinforced with sisal, flax, and jute fibers were investigated. The preparation characteristics of plaster composite were

thoroughly examined, and the tests were planned utilizing the CCD technique of RSM. In this study, the modeling and optimization of the flexural strength, displacement, and modulus are covered in depth [Boumaaza et al. 2021]. Benyettou et al. [2022] observed the effect and interaction of the input factors (cutting conditions) on the output parameters (delamination factor) during reinforced bio composites with date palm fibers (CDPF) drilling using RSM and ANOVA. In research employing hybrid abaca epoxy composites with red mud as the filler, RSM design was applied. Based on three components and three stages of complete factorial analysis, regression models were created for the flexural and impact strength of composite materials. Analysis was also done on response surface plots and interaction plots. ANOVA was used to analyze built-in models. It was found that the established regression models for composites' flexural and impact strengths were capable of accurately predicting their mechanical characteristics to within 90%. The two predominant parameters that have a significant impact on the flexural and impact strength of hybrid composite were the weight percentages of abaca and red mud. In other words, among the three input factors, red mud particle size had the least bearing [Sinha et al. 2019].

He et al. [2023] investigated the synergistic effects of two molding process parameters on the mechanical properties of jute reinforced polypropylene composites (jute/PP) from contour and surface plots through response surface methodology (RSM), with the goal of improving the mechanical properties of BFRCs and expanding their applications. ANOVA analysis was used to determine the importance of each molding process element on the mechanical behaviors of jute/PP. According to experimental results, multi-parameter co-optimization led to the best mechanical performance of molded jute/PP at 186.8°C, 5.6 MPa, and 10.1 min. This shows that the correlations between the mechanical properties and the molding parameters of jute/PP may be properly characterized by the response surface equations generated using RSM.

The fabrication of a coir fiber/carbon fiber/epoxy hybrid composite sheet was cut utilizing a pulsed CO_2 laser while employing RSM for modeling and parameter optimization in a different study. It has been determined that the existing second order response surface model is adequate. It is noted that for constructed models, the intersection of regression & linear, as well as square impacts of laser cutting variables, are significant

[Singh et al. 2021]. In another work, modified palm oil (MPO) and waste polyethylene terephthalate (WPET) were used to create bio-unsaturated polyester (BUP) composite. Using the response surface methodology, experimental circumstances' influencing factors were improved [Aydoğmuş et al. 2021]. Ghasemi et al. [2021] applied a Box-Behnken design of RSM with different wt.% of ethylene propylene dine monomer, grapheme Nano sheets and glass fiber to find the optimized parameters by analyzing desirability function

2.6 Summary of the Review

Summary of the review depicts that researcher are working tediously for some time to reduce the use of polymer and making them biodegradable without hurting their physico-mechanical properties. Natural fiber reinforced polymer composite proved to be one of the easiest ways to achieve that. Plastics has become part and parcel of our present life and if we can't discard it, we need ways to reduce it's bad impact on the environment. The best way to realize that potential is to conduct research by trial and error and logically experimenting. Natural fibers like coir, jute, rise husk, flux, pineapple leaf, banana leaf and stem, sugarcane bagasse, cotton and areca are some of the most harvested fibers on the planet which have been served us for ages. We have previously ignored it's use in composite manufacturing not only for biodegradation but also for reinforcing those composites for different applications. Recently these fibers are being used as reinforcing agent and sometimes being hybridized with other fibers and matrix to optimize their physical, mechanical, thermal and chemical properties.

However, inadequate study has been done on using cotton and areca fiber as reinforcing agents in thermoplastic matrices like polypropylene. Review of the experimental research reveals that inadequate information has been provided regarding the effect of alkali treatment, the incorporation of fiber volume into the matrix, and the percentage of various fibers in fiber volume. The impacts of these input parameters on various response characteristics, such as tensile strength, young modulus, flexural strength, flexural modulus, impact strength, hardness, and moisture absorption, among others, must be determined via further study.

2.7 Objectives of the Present Work

The objectives of the present investigation are:

i) Alkali treatment of areca and cotton fiber in different alkali solution to remove the lignin, hemicellulose, wax and oil from the surface of the fiber for increasing fiber's compatibility with polymer matrices.

ii) Manufacture of areca and cotton fiber reinforced polypropylene composite with hot press machine with different fiber loading and different ratio of areca and cotton fibers to ensure the biodegradability of the composite.

iii) Experimental investigation of different mechanical, thermal, chemical and physical properties of the manufactured composite for mechanical characterization for both treated and untreated fiber.

iv) Optimization of fiber loading, ratio and alkali percentage using response surface methodology for best mechanical properties.

2.8 Scope of the Thesis

Literature review narrates that cotton and areca fibers can be good reinforcing agent after being chemically treated specially alkali treated for a polymer composite. The reinforcement can definitely increase some mechanical properties and physical properties and can be used in household, automotive and industries for several use. The main objective of this work is to fabricate a novel hybrid composite and characterization of different physico mechanical properties and find out the optimum combination for fiber loading, ratio of the consisting fibers and concentration of alkali treatment by using RSM.

Chapter-1 describes composite materials, various matrix materials that may be utilized in a fiber reinforced polymer, in brief. It also emphasizes the development of natural fiber reinforced polymer and the variety of uses.

Chapter-2 gives us a glimpse of the prior research that has been done on various natural fibers reinforced composites using various production processes and characterizations of various qualities for different purposes. It also gives us ideas for future research in those areas.

Chapter-3 presents the method of fiber extraction, fiber preparation by alkali treatment and fabrication of cotton and areca fiber reinforced polypropylene composite by using a hot press device.

Chapter-4 provides an experimental inquiry in which various mechanical, physical, thermal, and chemical properties are examined using various tools inside various experimental setups. Graphical representations are frequently used to show the findings of experiments.

Chapter-5 presents the theory of response surface methodology (RSM) and modeling of tensile strength, tensile modulus, flexural strength, flexural modulus, impact strength, hardness and moisture absorption using RSM by varying input parameters such as fiber loading, ratio of cotton and areca fiber and alkali concentration. Optimized input parameters are also depicted by using desirability function.

Chapter-6 provides a through description and analysis of experimental findings for all mechanical, physical, chemical, and thermal characteristics and demonstrates the model's correctness by contrasting it with the findings of the experiment. Finally, a summary of the key contributions, suggestions for further research, and references are given.

Chapter-3

Material Preparation

3.1 Alkali Treatment of Areca and Cotton Fiber

Natural fibers undergo alkaline treatment to remove lignin, wax, pectin, oil, and hemicellulose. Additionally, the chemical structures of natural fibers' hydrogen bonds are also impacted by alkaline treatment, increasing the roughness of the fiber surface. Normally, the natural fiber is submerged in an alkaline solution, such as sodium hydroxide, for a set amount of time at a set temperature. It has been shown that soaking natural fibers in an alkaline solution increases their compatibility with the matrix as well [Magagula et al. 2022].

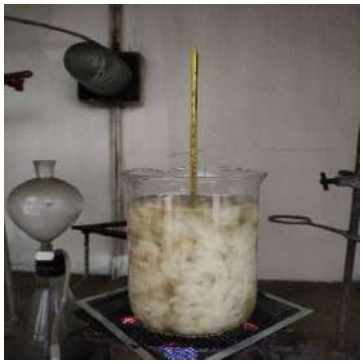
In this research the materials used were waste cotton fiber and areca husk fiber as reinforcement and polypropylene as matrix. Waste cotton fibers are generated from garments and cotton industry. They are the leftovers that are normally used in beds and mattresses as filler materials. As they are by products of cotton, they are a lot cheaper than pure cotton and easy to collect from local bedding stores. One kilogram of waste cotton was bought from a local bedding. Areca husk fiber was collected from the local betel leaf selling shops where both the leaf and nuts are sold together. In south east Asia people love to eat betel leaf and betel nut also known as areca nut together. In most cases the areca husk is thrown out as garbage and in this present work those thrown out areca husk will be used with cotton fiber as reinforcement to make hybrid polypropylene composite. Industrial grade six kilograms of polypropylene granule of Korea Ulsan PP Company Ltd. was purchased from a local supplier.

Before alkali treatment areca fibers were washed with tap water and then sun dried to washout the dust. NaOH was purchased and weighted with the help of an electronic precision balance to make sure 1%, 5% and 9% concentration. Right amount of

NaOH granule were mixed with 1 liter of distilled water in a beaker and wait some minute as the chemical is very reactive and instantly increases the temperature of water. Then areca and cotton fibers were submerged in alkali solution in different beakers and was heated at 70^{o}C for about two and half hours and they were occasionally starred to disperse the heat evenly. Eventually, tap water and distilled water were used to wash the fibers for at least 20 minutes to thoroughly remove the NaOH. To fully evaporate the moisture, the fibers that contained water were placed in the oven [Rahman et al. 2018]. Fig 3.1 depicts the alkali treatment process.

Untreated cotton fiber

Untreated areca fiber



Cotton fiber treatment

Areca fiber treatment

Washing with distill water

Fully treated fiber

Fig. 3.1 Photographic view of alkali treatment of cotton and areca fiber

3.2 Manufacture of Areca and Cotton Fiber Reinforced Composite

Multiple reinforcing agents are used in the same polymer matrix to create hybrid composites, which are intended to improve the composite's qualities. The effects of the components might be either synergistic or antagonistic when many reinforcing compounds are combined. Additionally, the qualities may be adjusted by contrasting the advantages of two different materials. A wider range of qualities that are not possible with single fiber

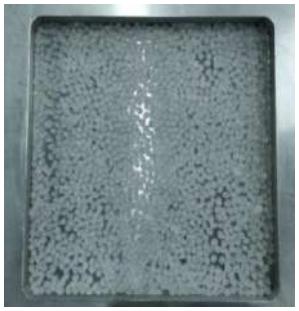
reinforced composites are offered by the use of numerous reinforcements in a matrix. As a result, the scientific community has begun to pay much attention to the hybridization of reinforcing agents with natural and synthetic origins.

In the current study, the percentages of reinforcing fiber load, cotton and areca fiber load, and percentage of NaOH concentration have been varied to observe changes in the physico-mechanical characteristics of the novel hybrid composite. Both cotton and areca fiber were treated in 1%, 5% and 9% alkali solution. 5% ,10% and 15% fiber load was used to reinforce the polypropylene matrix varying the percentage of cotton and areca from 25% to 75% in the total fiber load. In total of 16 composites were manufactured using local hot press machine which is very cheap compared to the hot press machine in different laboratories. A 150×150×4 mm^3 mould was made with aluminum.

First, cotton and areca fiber that had been treated and dried were weighed on a precision scale to determine the proper proportion of fiber loading. The fibers were then trimmed using scissors into 2 to 5 mm in lengths. To guarantee the created composite was removed smoothly, silicon spray was sprayed on the lower portion of the aluminum mold. The mould was then filled with one layer of polypropylene granules. After that, a layer of manually mixed cotton and areca fiber was positioned in a random orientation on top of the first layer. The final polypropylene layer was then stacked on top of the fiber layer. The upper mould was then securely fitted over the three layers after being coated with silicon spray. Then, using a screw mechanism, the higher die of the hot press machine is progressively lowered and pressed the upper mould as much as possible while the mold is retained over the lower die. The local hot press machine operates on a 440 V connection. In order to heat both the dies and everything in between, it features two 1000 wat coils in the upper and lower die. Each die weighed around 40 kg and was constructed of cast iron. The dies need to heat up adequately for roughly an hour. It is prepared for use once the dies have been preheated for at least one hour. If the dies are properly heated up this local hot press machine may produce a composite in around 20 to 30 minutes. The ideal moment to remove the composite from the mould was identified after some trial and error. Fig 3.2 shows the mould preparation with fiber and matrix. Fig 3.3 shows the set up for composite manufacturing with hot press machine as well as the fabricated composite.

Treated cotton fiber

Treated areca fiber



1st layer of PP

2nd layer of fiber mix

3rd layer of PP

Upper and lower mould

Fig: 3.2 Photographic view of mould preparation with matrix and fibers

Hot press machine

Manufactured composite

Fig: 3.3 Photographic view of hot press machine and manufactured composite

A temperature of about 165ºC causes polypropylene to begin melting. The upper die is lowered using the die's screw wheel mechanism after the first ten minutes, after the fiber and polypropylene mold has heated up completely. This allows the upper mold to be pressed. The excess molten polypropylene now begins to flow out of the mould clearance as both layers of polypropylene start to melt at this time, making it easy for the top die to press the mold. This helps the mold to retain 150mm long, 150mm wide, and 4mm high hybrid composite in between the upper and lower mould. The hot press machine is switched off after 20 to 25 minutes and allowed to cool for 10 minutes before the top die is gradually taken from the mould using the screw wheel mechanism. This process is repeated until all of the surplus polypropylene has been removed from the mold. Once the mold has been removed from the hot press machine, the heat is dissipated by submerging it in cold water. After removing either the top or lower mould from the other, we are left with a hybrid composite. Using this novel method, 16 hybrid composites were created. These composites were then removed from the mould and the excess polypropylene was shredded with a scissor or an anti-cutter.

Chapter-4

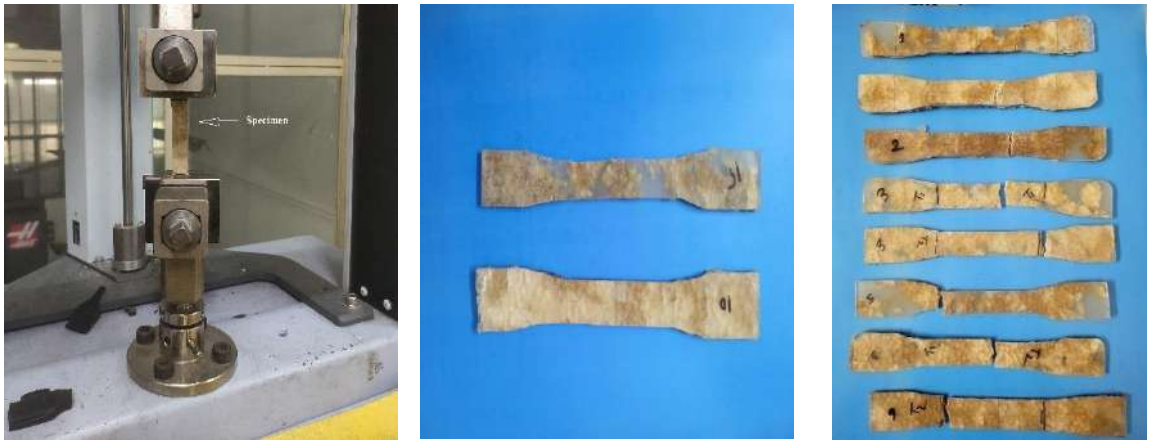
Experimental Investigation

4.1 Mechanical Characteristics Testing Procedure

Numerous studies have been conducted to comprehend the mechanical, physical thermal and chemical behavior of composite materials and create design techniques that maximize their qualities. Mechanical qualities play a crucial role in the use of these composite materials, and studies constantly seek to describe new composites by determining their mechanical properties in various combinations. After determining how input parameter variations affect mechanical qualities, mathematical modeling may be used to forecast various outcomes and optimize the optimum input parameter combination to produce the most balanced composite.

In this study, a wide range of mechanical properties have been investigated, inclu ding tensile strength, flexural strength, tensile modulus, flexural modulus, impact strength, hardness, and moisture absorption (physical property). Specimen of different size and shape was prepared for different types of tests. Fifteen composites with different combinations of fiber load, percentage of cotton and areca fibers in fiber load and percentage alkali concentration was tested as well as one untreated composite.

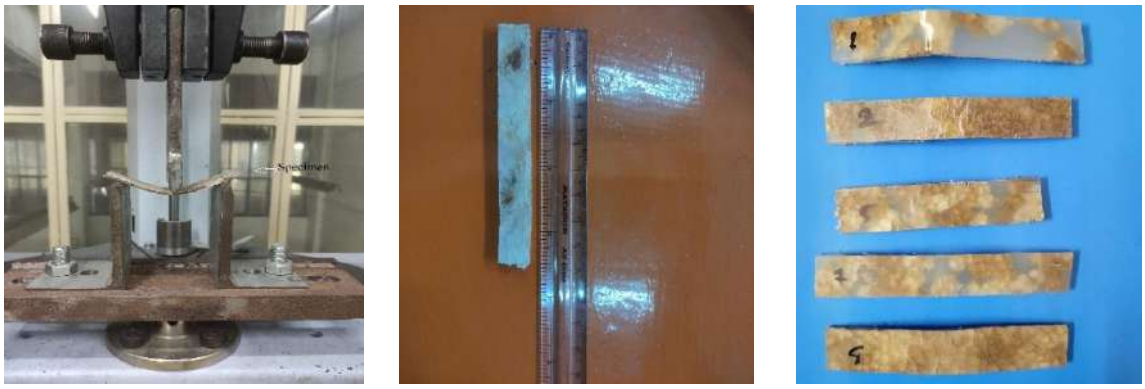
Specimens were cut into the shape of dog bone with a gauge length of 57 mm. Tensile tests were performed using a universal testing machine (UTM), model Autograph X series (maximum capacity 100 KN) in accordance with ASTM D 638-01. Until tensile failure, each test was run. Cross head speed was 5mm/min. The test method's determination of tensile properties aids in determining the stress of the composite specimen, its features of fracture under tensile load, and the load-strain behavior bearing qualities of the fiber and matrix materials. Fig 4.1 shows the tensile test procedure.



Tensile test with UTM Specimen before test Specimen after test

Fig: 4.1 Photographic image of tensile testing and specimens

The three-point bend test is generally performed on composites to determine flexural strength. The depth of support span shall be 16 times the depth of the beam. Specimen width shall not exceed one fourth of the support span for specimens greater than 3.2 mm in depth. Total length of the specimen should be span length plus 25 mm extra length. Flexural test was conducted with the help of same Universal testing machine by changing the settings and setup. Specimens were prepared according to ASTM D 790-00. All the specimens were individually measured with slide calipers and the machine were calibrated. The cross-head speed was 5mm/min. Fig 4.2 shows the flexural test procedures.

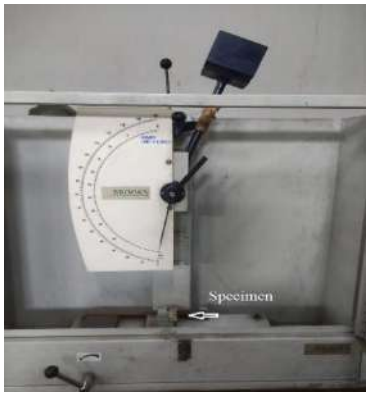


Flexural test with UTM Specimen before test Specimen after test

Fig: 4.2 Photographic view of flexural testing and specimen

For Charpy impact test, specimen was cut according to ASTM A370 with the help of BROOKS impact tester (Max impact energy 50 joule). The pointer of impact test machine was set to zero degree (50 joule). The specimen of dimension 55 mm × 4mm × 14mm was placed in the holder for impact tester with a v notch cut in the middle. Then the hammer of the tester was released. The final position of the pointer was observed which

indicates the absorbed energy(J) before fracture. Figure 4.3 shows the impact test procedure.



Charpy Impact tester


Specimen before test


Specimen after test

Fig: 4.3 Photographic image of Charpy impact test

The ability of a substance to resist being permanently indented is known as hardness. Results for thermoplastic materials are often presented as readings on a R scale or M scale. The R scale specifies the use of an indenting ball with a 0.50 in. (12.70 mm) diameter and a main load of 60.00 kg. The primary load for the M scale is 100.00 kg, and the ball has a diameter of 0.25 in. (6.35 mm). A material's resistance to permanent indentation is one way to describe its hardness. Shore hardness of D scale is also used to measure hardness of plastics [Campo 2008]. In this present study, Rockwell hardness R scale was used to measure the hardness. Fig 4.4 shows the procedure of hardness test.

Fig: 4.4 Hardness testing with Rockwell hardness tester (R scale)

Natural fibers are resilient, yet depending on the extraction procedure and medium, they might exhibit varied properties when exposed to moisture. varied natural fibers exhibit varied moisture absorption rates and post-moisture behavior. Moisture

absorption of cotton-areca reinforced polypropylene hybrid composite were observed (ASTM D 570) after submerging the specimens for 24 hours in distill water and then measuring how much weight they gained. Initially dry specimen was weighed to find out the weighed difference between dry and wet samples. Fig 4.5 shows moisture absorption test procedure

Submerged specimen weighing wet specimen

Fig: 4.5 Photographic view of moisture absorption test

4.2 Physical, Chemical and Thermal Properties Testing

By creating an infrared absorption spectrum, Fourier Transform Infrared Spectroscopy (FTIR) can identify the chemical bonds in a molecule. The spectra provide a sample profile, or specific chemical fingerprint, that may be used to screen and scan samples for a variety of components. For analyzing minerals, polymers, corrosion byproducts, and unidentified compounds, X-ray diffraction is helpful. Identification and measurement of the crystalline phases, estimation of the crystallinity percentage, and study of the crystal structure are some of the most popular tests. Thermogravimetric Analysis (TGA) is a method for analyzing materials that involves heating a sample under controlled conditions and measuring its mass as a function of temperature or time. The temperature and weight loss are measured as the material's components volatilize over time. SEM, or scanning electron microscopy, creates high resolution photographs of an item by scanning its surface in order to make detailed, enlarged images of it. SEM does this utilizing a laser-focused electron beam. Information about the object's composition and physical characteristics may be seen in the photos that are produced. Fig 4.6 shows the setup used for FTIR, XRD, SEM and TGA-DSC.

FTIR analysis machine

XRD analysis machine

TGA-DSC testing machine

SEM analysis machine

Fig: 4.6 Photographic view of FTIR, XRD, TGA-DSC and SEM test machine

4.3 Experimental Result

Composites were fabricated by following RSM designed combination by varying fiber load, percentage of cotton and areca and percentage of NaOH concentration.15 composites were manufactured for modeling and one more untreated composite was manufactured to compare it with the treated composites. Tensile strength, tensile modulus, flexural strength, flexural modulus, impact strength, hardness and moisture absorption were measured for all 16 composites for characterization and comparison and later making a prediction and optimization model. Tensile strength was directly measured from the machine by using eqn. (4.1),

$$\text{Tensile Strength } (T_s) = \frac{\text{Maximum Force}}{\text{Initial Area}} \text{ (MPa)} \tag{4.1}$$

Tensile modulus or young modulus was calculated by taking the slope of stress strain curve generated by the ultimate tensile machine. Flexural strength was directly calculated by the same machine using following eqn. (4.2),

$$\sigma_f = \frac{3PL}{2bd^2} \tag{4.2}$$

Where,

σ_f=Stress in the outer fiber at mid span, MPa

P=Load, N

L=Support span, mm

b =width of beam, mm

d= Depth of beam, mm

Flexural Modulus was calculated by using the eqn. 4.3 where m is the slope derived from the stress stain curve

$$E_B = \frac{L^3 m}{4bd^3} \tag{4.3}$$

Impact energy(J) is directly measured from the Charpy impact tester and is divided by the notch area to get the impact strength in KJ/m^2. For water absorption test, dry samples were initially weighed in a precision balance and then wet sample were weighed after submerging for 24 hours in water and removing the excess water with dry cloths. The water absorption% is calculated from eqn. (4.4),

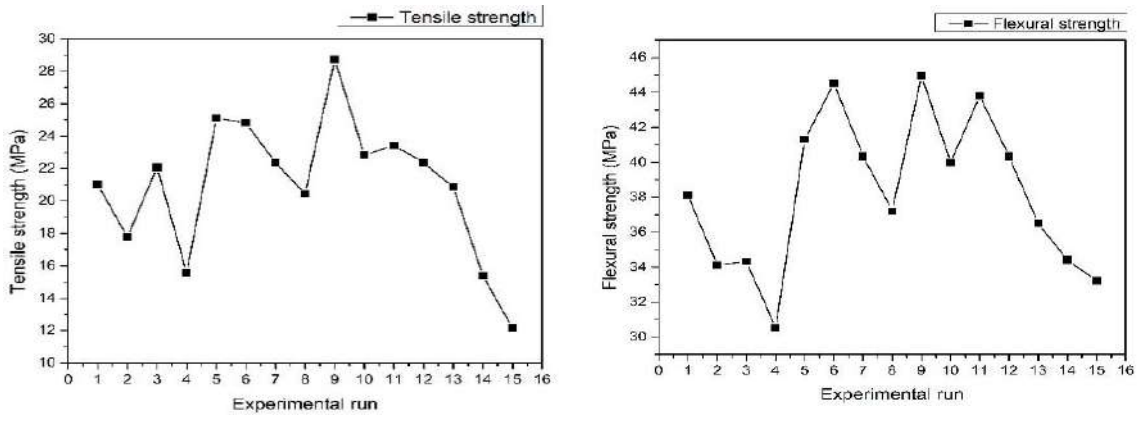
$$\text{Water absorption\%} = \frac{\text{Final Weight} - \text{Initial Weight}}{\text{Initial Weight}} \times 100 \tag{4.4}$$

Hardness was directly measured from the gauge of R scale. Table 4.1 shows the RSM designed combination for manufacturing 15 different hybrid composite varying 3 input parameters, fiber load, cotton and areca % in the fiber load and percentage of NaOH.

Table 4.1 RSM combination for experimental run

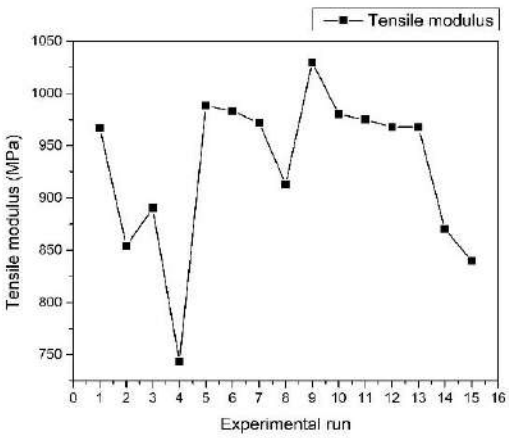
Experiment Run	Fiber load (%)	Cotton and areca (%)	NaOH (%)
1	10	75%+25%	9
2	15	25%+75%	5
3	10	25%+75%	9
4	15	50%+50%	9
5	5	50%+50%	1
6	5	25%+75%	5
7	10	50%+50%	5
8	10	25%+75%	1
9	5	75%+25%	5
10	10	50%+50%	5
11	5	50%+50%	9
12	10	50%+50%	5
13	10	75%+25%	1
14	15	75%+25%	5
15	15	50%+50%	1

Fig 4.7 shows values of tensile strength, flexural strength, and Fig 4.8 shows tensile modulus, flexural modulus, impact strength, hardness and moisture absorption against experimental run.

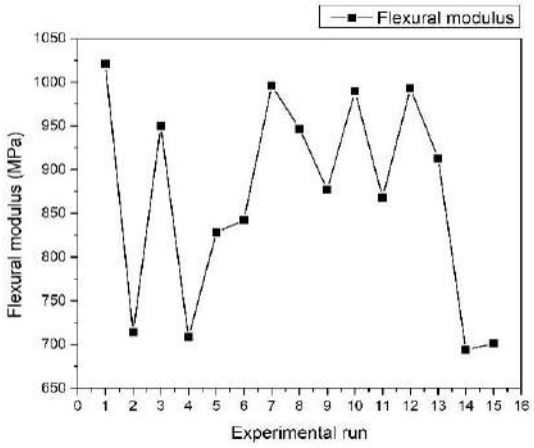


Tensile strength Flexural strength

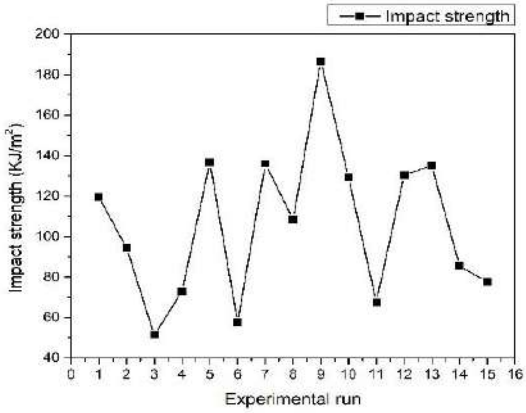
Fig: 4.7 Variation of mechanical properties (Tensile and Flexural strength)



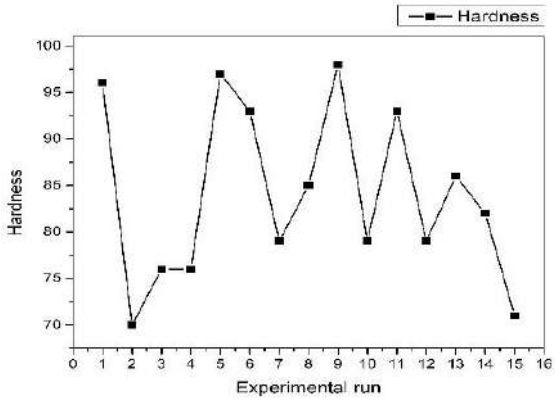
Tensile modulus



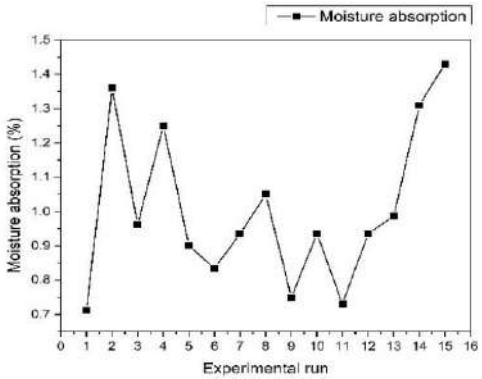
Flexural modulus



Impact strength



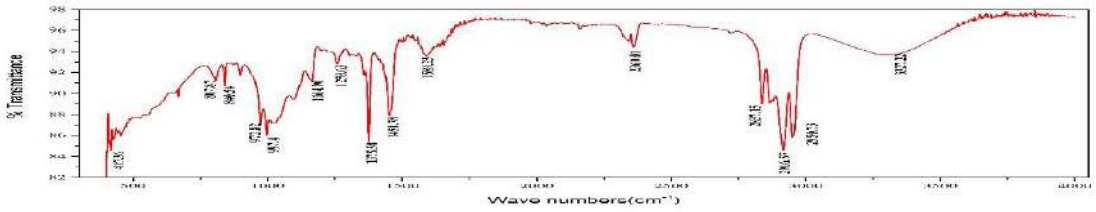
Hardness



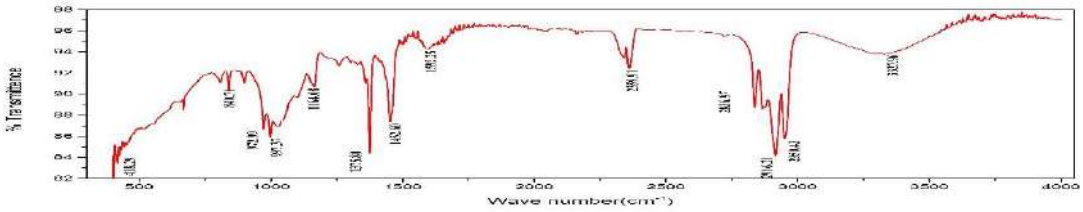
Moisture absorption

Fig: 4.8 Variation of tensile modulus, flexural modulus, impact strength hardness and moisture absorption with experimental run

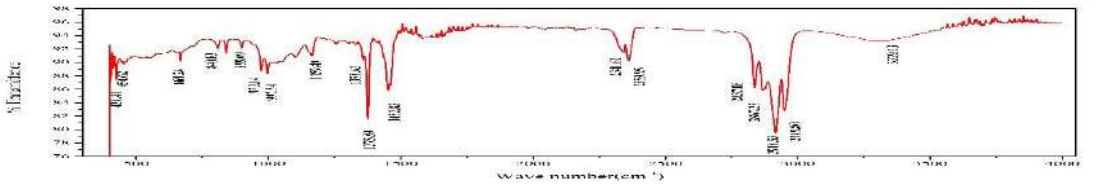
All these 7 responses were also measured for one untreated sample with 5% fiber load, 75% cotton and 25% areca and 5% alkali concentration to see the effect of treatment on fibers. Tensile strength 19.9MPa, flexural strength 37.98MPa, impact strength 100.3KJ/m^2, hardness 84, tensile modulus 910MPa, flexural modulus 810 MPa and moisture absorption 2.13% were measured from the test.



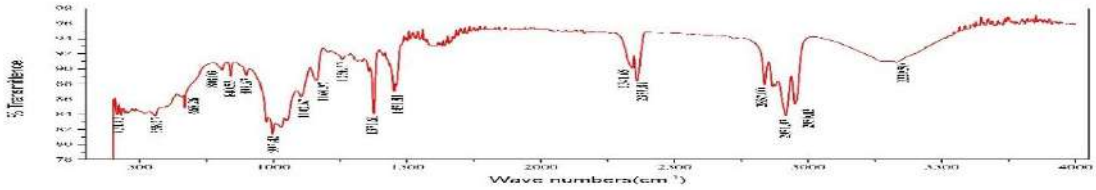
Experiment run 5



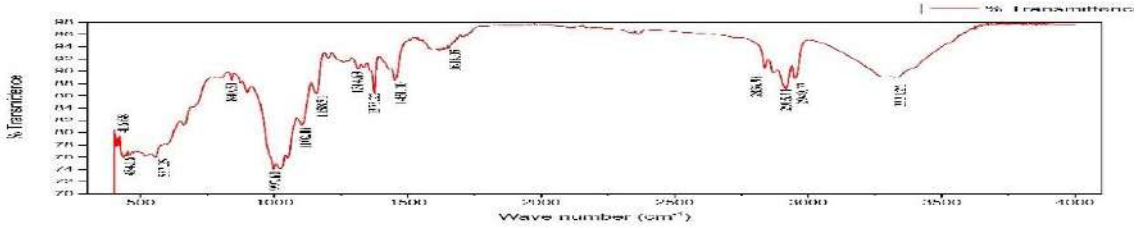
Experiment run 6



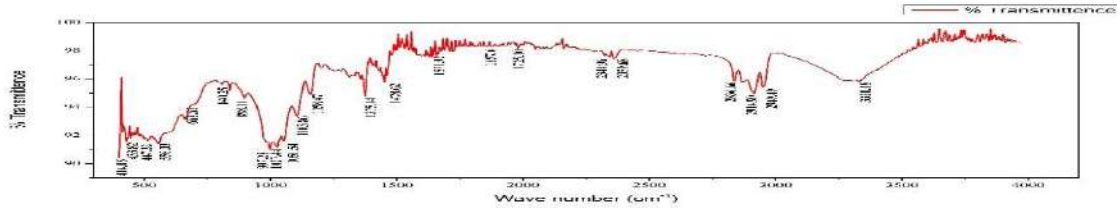
Experiment run 9



Experiment run 4



Experiment run 1



Untreated

Fig: 4.9 Photographic view of FTIR spectrum of different test run

Fourier transform infrared Spectroscopy (FTIR) was observed for 6 sample. From table 4.1, Experiment run 5, 6 ,9, 4, 1 and untreated composites were chosen to investigate the variation caused by NaOH concentration and fiber loading and ratio of cotton and areca in fiber loading. Fig 4.9 shows the FTIR spectrum of the aforementioned 6 run.

Xray diffraction was observed for experiment run 16(untreated), 5, 9, 10 and 4(table 4.1), total of 5 specimens copper K-α was used as x-ray energy with a 2θ range from 10 to 80 degree. Rectangular specimens of 30mm length and 19mm width and 4 mm depth were provided. XRD data was plotted in origin pro software and baseline was corrected for better visualization as well as calculating crystalline area and amorphas area for determining crystallinity index. Peaks were also plotted using origin pro software. First the area of the major crystalline peaks was calculated. The areas of all the crystalline peaks were added to establish the overall crystalline phase. Simply combining the areas of all the peaks produced the total area of the crystalline and amorphous phases. By dividing the entire area of the crystalline phase by the combined area of the crystalline and amorphous phase, the relative percentage crystallinity was calculated [Mannan et al. 2006]. The area of crystalline peaks and amorphas peaks were calculated by integration of two points using the origin pro software. The hybrid composite's crystal size was determined using Scherrer's equation in (4.5).

$$CS = \frac{K\lambda}{\beta Cos\theta} \tag{4.5}$$

Here λ is The wave length of the radiation, the full width at half maximum (FWHM) of the peaks, θ is the Bragg's diffraction angle, and K = 0.89 make up the Scherrer's constant. Table 4.2 shows the result of crystallinity index and crystal size of the 5 specimens and Fig 4.10 shows the X-ray diffractogram of the 5 specimens with their distinct peak.

Table: 4.2 Crystallinity index and crystal size of 5 specimens

Specimen	Fiber load %	Cotton and areca%	NaOH %	Crystallinity index %	Crystal size, nm
1	5	75%+25%	untreated	64.73	9.43
2	5	50%+50%	1	67.19	10.19
3	5	75%+25%	5	68.46	10.18
4	10	50%+50%	5	71.41	10.16
5	15	50%+50%	9	72.73	12.00

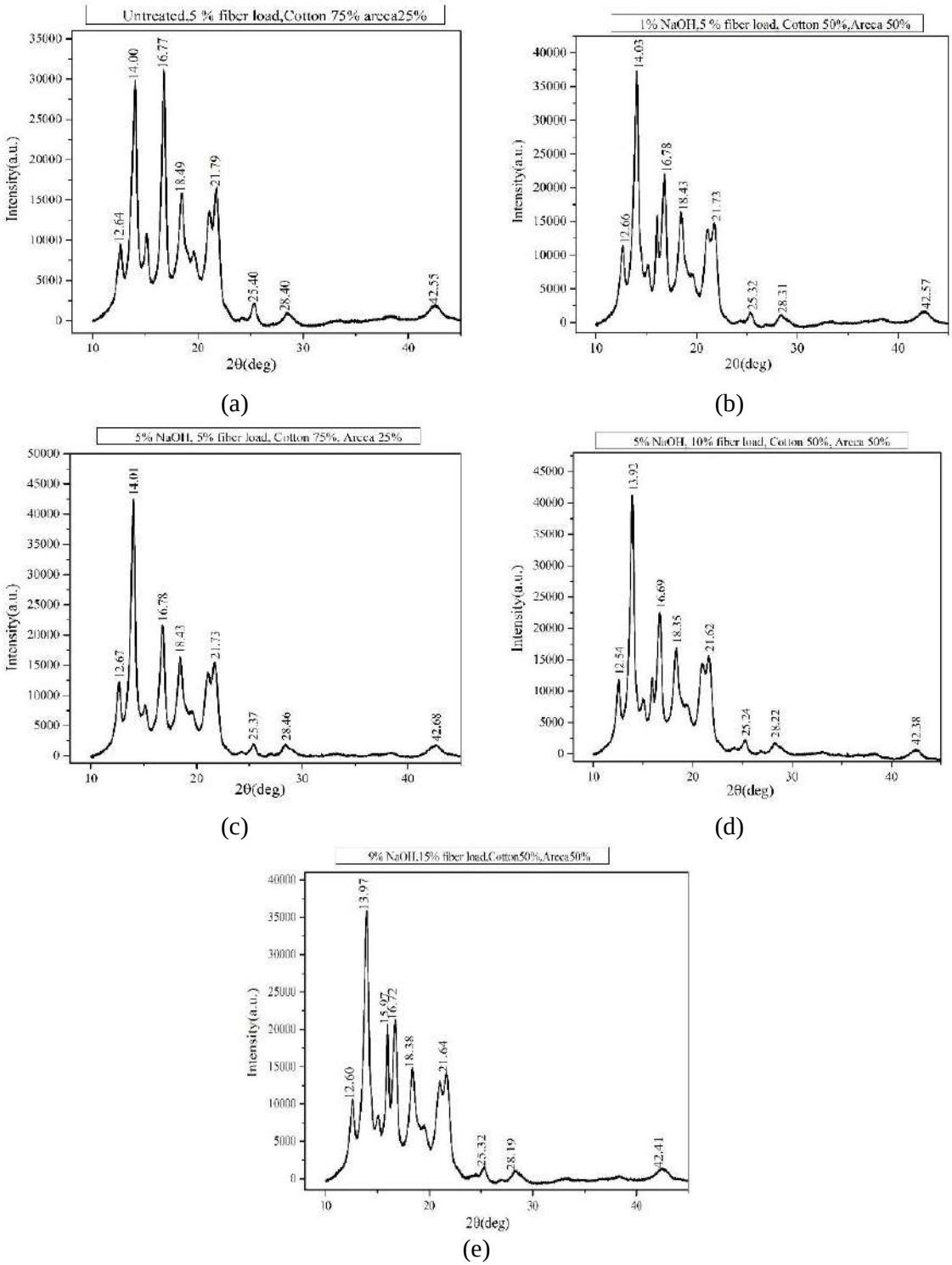
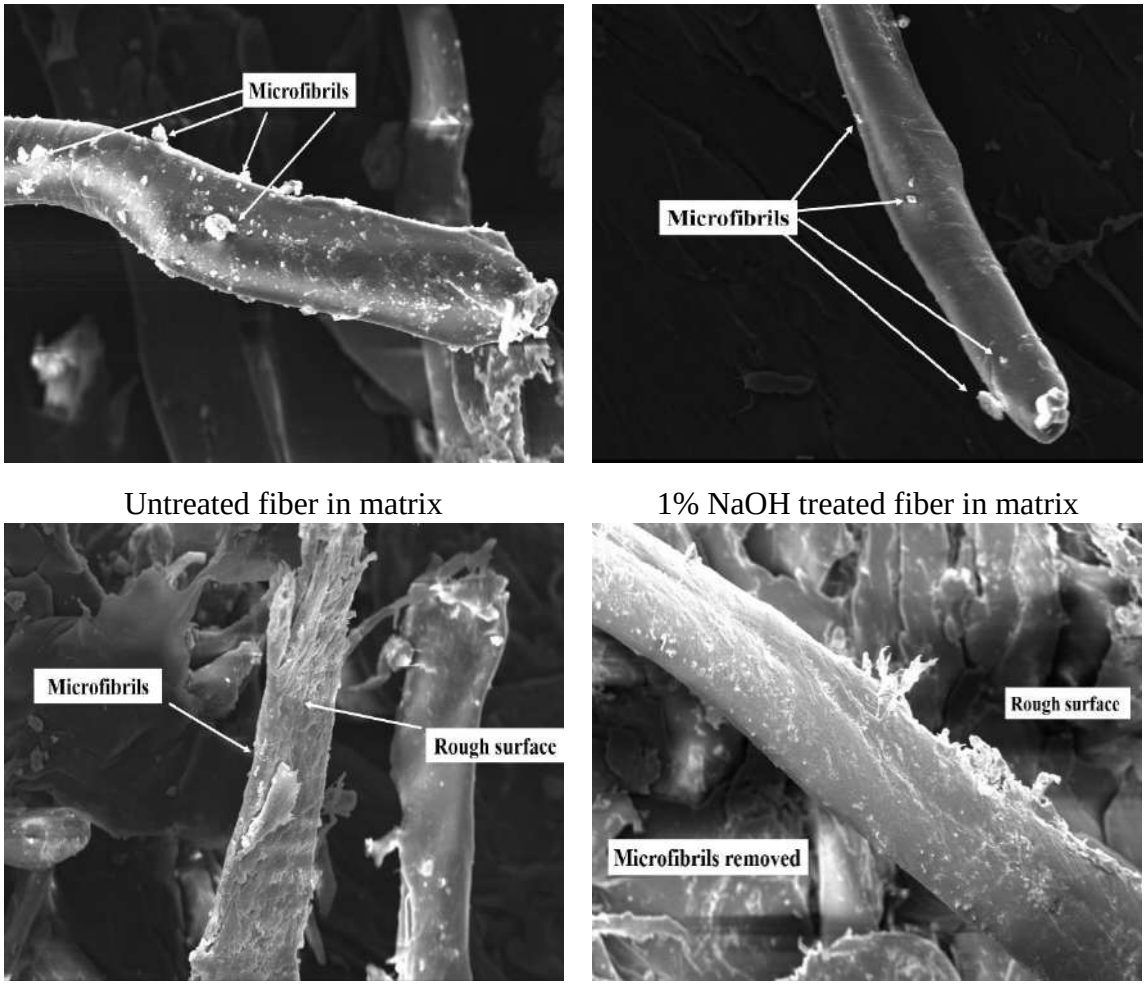


Fig: 4.10 Photographic view of XRD peak of 5 specimens

Scanning electron microscopy (SEM) image was investigated to see the effects of untreated, 1%, 5% and 9% alkali treated fiber's surface morphology in the polymer composite. Fig 4.11 shows SEM images



Untreated fiber in matrix

1% NaOH treated fiber in matrix

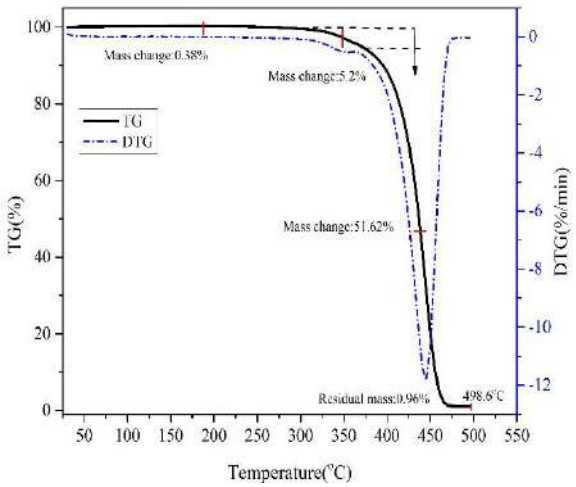
5% NaOH treated fiber in matrix

9% NaOH treated fiber in matrix

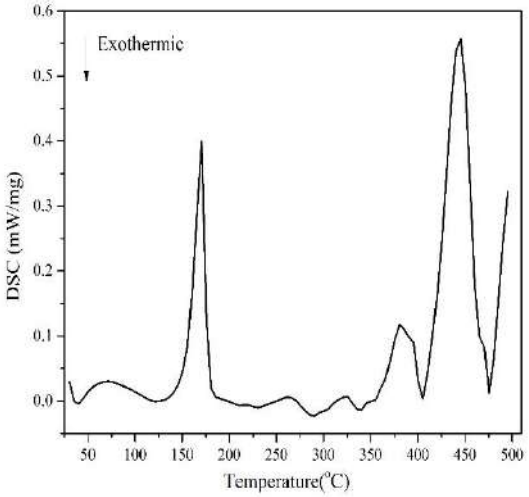
Fig: 4.11 SEM image of untreated and treated fibers

Thermogravimetric analysis (TGA), differential thermogravimetry (DTG) and differential scanning calorimetry (DSC) was done for Specimen 3 and 5 of table 4.2 to determine the composite's thermal stability and different transition temperature by observing the weight loss of the specimens when it was subjected to a controlled temperature program in an inert Nitrogen gas environment as well as how much heat was being absorbed into the specimen as a function of time. With the help of the simultaneous thermal analyzer "NETZSCH STA 449 F3 Jupiter" from a temperature range of 30 to 500, mass change and heat absorption were observed. The heating and cooling rate was set at

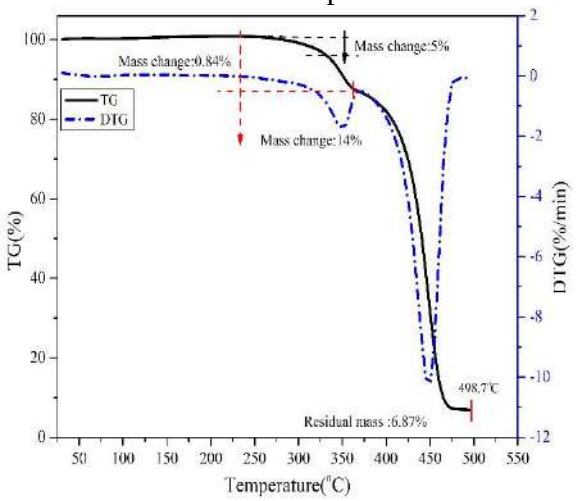
10^{o}C per minute and the specimens were kept in a Al_2O_3 crucible. Fig 4.12 shows TGA-DTG, and DSC graph of the 2 specimens.



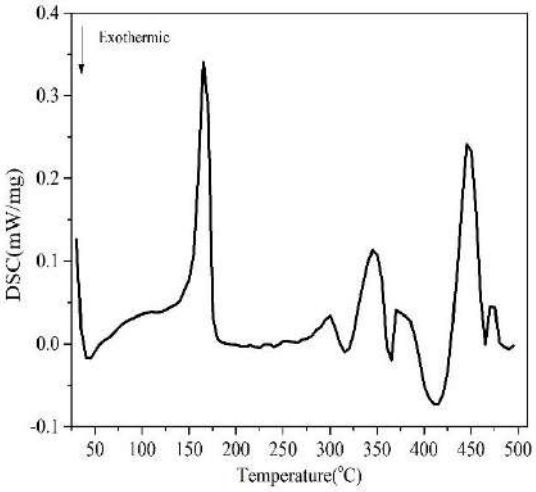
TG-DTG curve of specimen no 3



DSC curve of specimen no 3



TG-DTG curve of specimen no 5



DSC curve of specimen no 5

Fig: 4.12 Photographic view of TG-DTG and DSC curve

Chapter-5

Mathematical Modeling

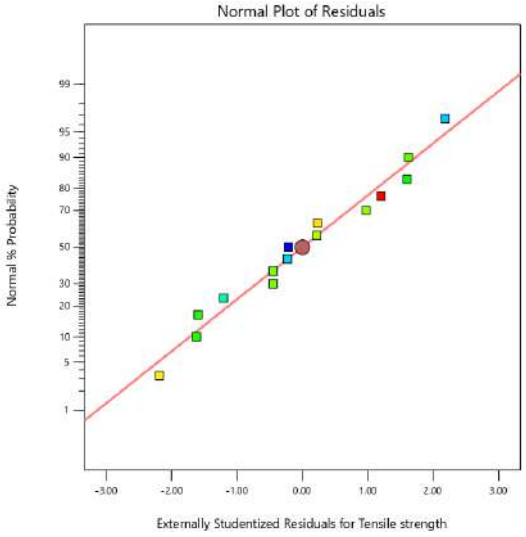
5.1 Modeling of Mechanical Properties by RSM

The response surface methodology (RSM) is one of the designs of experiment (DOE) methodologies for optimizing the operational parameters or conditions of a system as opposed to the conventional approach, which cannot be optimized. When many different possible input factors affect a process's performance or a product's feature, RSM is very helpful in comparison to previous DOEs, it may also assess the interactions between various factors and their impacts on one or more response variables. Test are run at a certain order following the DOE by changing some input parameters to monitor the change in output response. Errors in the entire process might have come from a few different places. It is possible for errors to occur while manually collecting data from a device. The measuring tool itself may also be inaccurate. Numerical errors including data uncertainty, round-off errors, truncation errors, and a few more might arise when data is acquired with the use of a computer. The mistake in RSM is thought to be spread randomly. The response function must be approximated by a first or second order model. The primary goal is to establish the statistical significance of the factors associated with a specific response through goodness of fit and to establish the ideal settings within the higher or lower level of control variables to limit or maximize the response of interest [Khuri and Mukhopadhyay 2010]. The fitted second-order polynomial regression model, often known as the quadratic model, is used to suggest the output response. The following is how the quadratic model of Y may be expressed in eqn. (5.1) and (5.2),

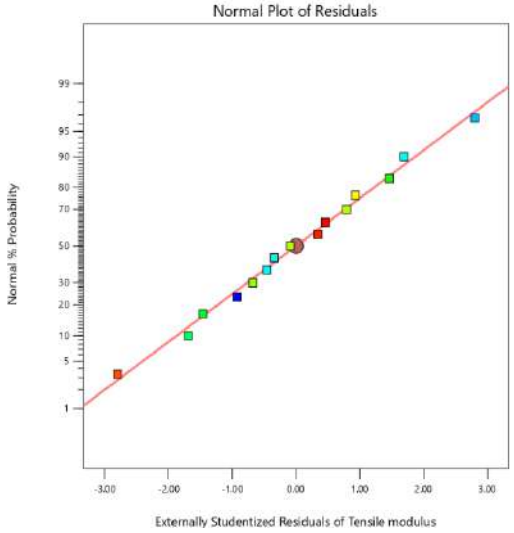
$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \varepsilon \tag{5.1}$$

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{ij=1}^{k} \beta_{ij} X_i X_j + \varepsilon \tag{5.2}$$

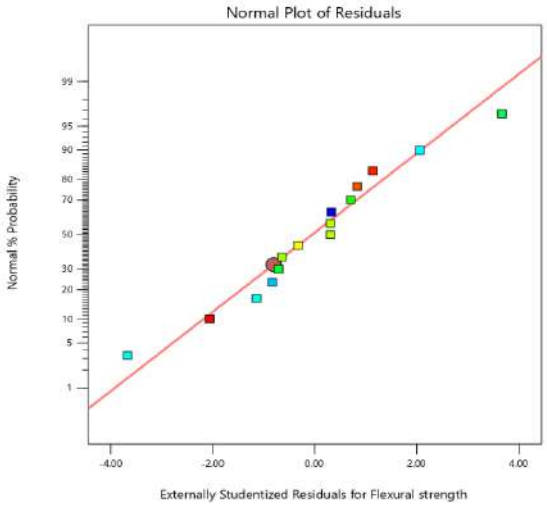
Here, the response (Y) estimation is based on the quadratic model with interactions as equation 2 where βo, βi, βii and βij represent the regression coefficients of the linear, quadratic and interaction terms, respectively. X_i represents input variable, fiber load percentage (F_L), percentage of cotton and areca in fiber load ($P_{L)}$ and concentration of NaOH for fiber treatment ($A_{\%}$). Normal probability graphs are plotted in fig 5.1 to test the effectiveness of the model and to show that models have good agreement with the added data. The normal probability plot of residuals may be used to graphically represent the model efficiency. The residuals must obey a normal law in order to pass this test. Figures show that the model residuals roughly trace straight lines, satisfying the normality.



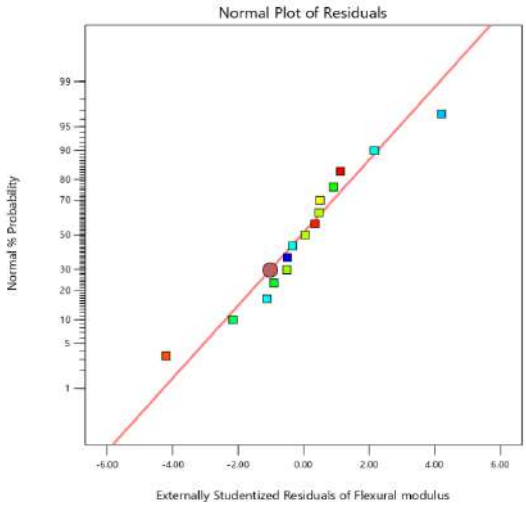
Tensile strength



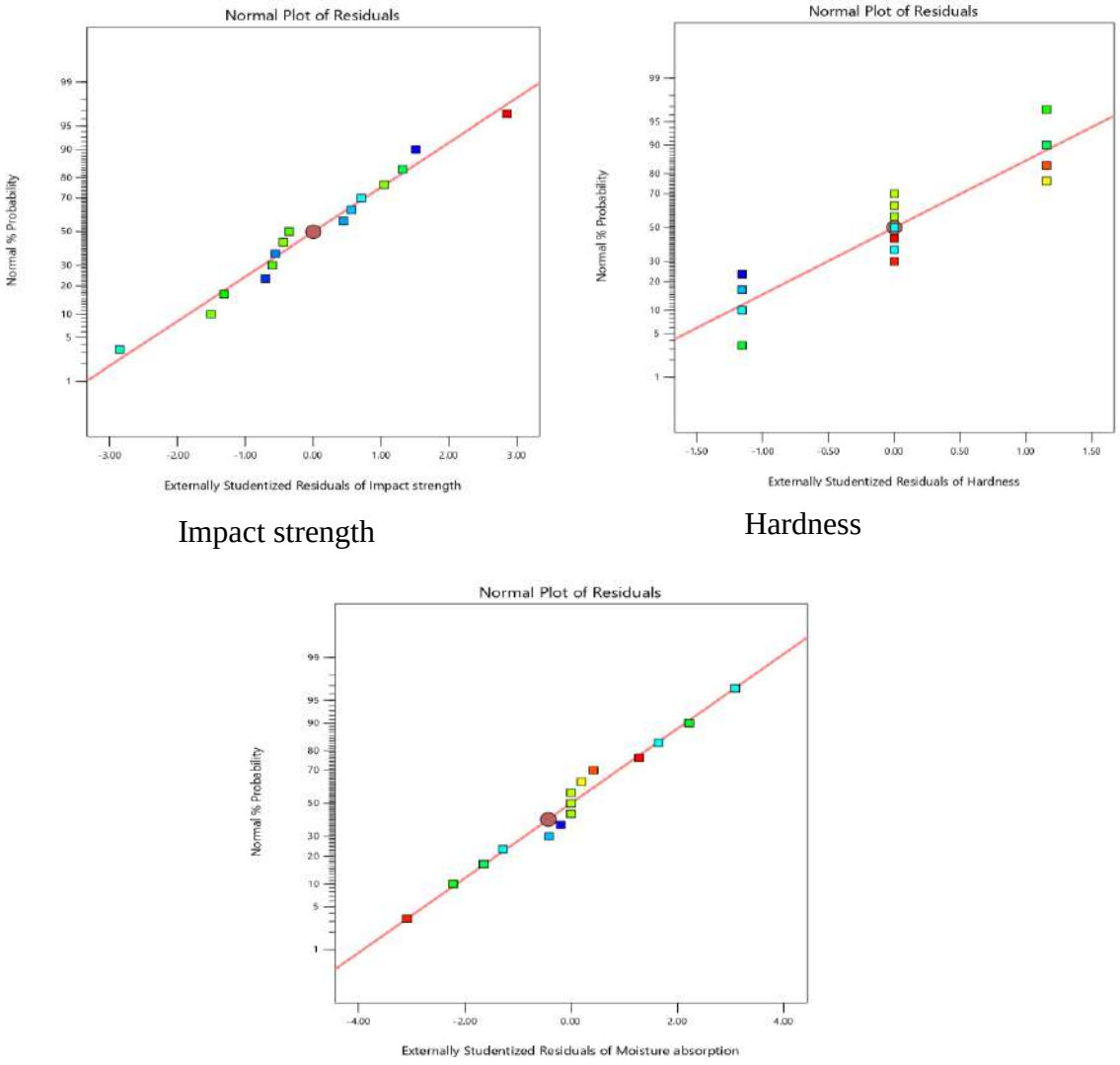
Tensile modulus



Flexural strength



Flexural modulus



Impact strength

Hardness

Moisture absorption

Fig: 5.1 Residual plot for different mechanical and physical properties

By using Box-Behnken design(BBD) with 15 experimental run by varying input parameters as Fiber load percentage (F_L), percentage of cotton and areca(only cotton percentage are shown in fig 5.2-5.8 and if we subtract it from 1, we will get the areca percentage) in fiber load (P_L) and concentration of NaOH for fiber treatment ($A_{\%}$) tensile strength(T_S), tensile modulus(T_M), flexural strength (F_S), flexural modulus(F_M), impact strength(I_S), hardness(H) and moisture absorption (M_A), all these 7 output response was observed .These input and output parameters are then used in Design Expert 12.0 RSM model to make a regression model that establishes the cause and effect between factors and outputs. By using the model, seven quadratic equations for tensile strength, tensile

modulus, flexural strength, flexural modulus, impact strength, hardness and moisture absorption (eqn. 5.3 to 5.9) are found.

$$T_S = 21.48 + 0.432\ F_L + 5.38\ P_L + 0.918\ A_\% - 0.05738\ F_L^2 + 9.57\ P_L^2 - 0.1259\ A_\%^2 - 1.264\ F_L P_L + 0.06363\ F_L A_\% - 0.370\ P_L A_\% \quad (5.3)$$

$$T_M = 789.5 + 32.70\ F_L + 195.4\ P_L + 32.14\ A_\% - 2.7467\ F_L^2 - 154.7\ P_L^2 - 2.589\ A_\%^2 - 2.40\ F_L P_L - 0.3375\ F_L A_\% + 1.75\ P_L A_\% \quad (5.4)$$

$$F_S = 41.90 - 0.697\ F_L + 7.95\ P_L + 1.901\ A_\% - 0.00105\ F_L^2 - 11.34\ P_L^2 - 0.1863\ A_\%^2 - 0.032\ F_L P_L - 0.0646\ F_L A_\% + 1.125\ P_L A_\% \quad (5.5)$$

$$F_M = 259.2 + 143.57\ F_L + 189.5\ P_L + 6.48\ A_\% - 7.766\ F_L^2 - 236.1\ P_L^2 - 1.266\ A_\%^2 - 4.20\ F_L P_L - 0.138\ F_L A_\% + 26.34\ P_L A_\% \quad (5.6)$$

$$I_S = -79.7 + 23.10\ F_L + 418.1\ P_L - 3.64\ A_\% - 0.817\ F_L^2 - 87.7\ P_L^2 - 1.421\ A_\%^2 - 27.50\ F_L P_L + 0.808\ F_L A_\% + 10.40\ P_L A_\% \quad (5.7)$$

$$H = 145.29 - 5.413\ F_L - 84.75\ P_L - 5.078\ A_\% + 0.1050\ F_L^2 + 66.00\ P_L^2 + 0.1641 A_\%^2 + 1.400\ F_L P_L + 0.1125\ F_L A_\% + 4.750\ P_L A_\% \quad (5.8)$$

$$M_A = 1.057 - 0.0608\ F_L + 0.112\ P_L - 0.0002\ A_\% + 0.005565\ F_L^2 - 0.174\ P_L^2 + 0.000242\ A_\%^2 + 0.0070\ F_L P_L - 0.000125\ F_L A_\% - 0.0465\ P_L A_\% \quad (5.9)$$

These equations can be used to predict the output variables by varying input variables. In each of the equation, some input variables have significant impact on the output variables and sometimes the square terms or the effect of interaction between 2 input variables can also have significant impact on this output response. To determine those significant variables and their interacting effect on the model, analysis of variance (ANOVA) is used. Sequential sum of squares, F-value, and P-value make up the ANOVA table. A factor's importance is shown by the P-value to a 95% level of confidence. A higher F-value denotes a factor's relative significance being higher. SS stands for the sum of squared deviations from the mean, while MS stands for mean square the variance related to each individual term. By dividing SS by degree of freedom (df), the variance is determined. For the aim of calculating the performance of the concerned model in depth, R squared (R^2), adjusted R squared (adj R^2), predicted R squared (pred R^2), and sufficient precision are determined. The coefficient of determination, or R^2, is a tool for assessing how well independent factors may predict the dependent variable. Models with higher R^2 values are better able to predict outcomes accurately [nagerkelke,1991]. Table 5.1 Shows the ANOVA table for Tensile strength (T_S)

Table: 5.1 ANOVA for tensile strength (T_S)

Source	Sum of squares	df	Mean Square	F-value	p-value	Remarks
Model	254.46	9	28.27	183.29	< 0.0001	significant
F_L	212.08	1	212.08	1374.82	< 0.0001	significant
P_L	0.1058	1	0.1058	0.6859	0.4453	not significant
$A_{\%}$	1.54	1	1.54	9.98	0.0251	significant
$F_L \times P_L$	9.99	1	9.99	64.73	0.0005	significant
$F_L \times A_{\%}$	6.48	1	6.48	41.99	0.0013	significant
$P_L \times A_{\%}$	0.5476	1	0.5476	3.55	0.1183	not significant
F_L^2	7.6	1	7.6	49.26	0.0009	significant
P_L^2	1.32	1	1.32	8.56	0.0328	significant
$A_{\%}^2$	14.99	1	14.99	97.14	0.0002	significant
Residual	0.7713	5	0.1543			
Lack of Fit	0.624	3	0.208	2.82	0.2723	not significant
Pure Error	0.1473	2	0.0736			
Total	255.23	14				

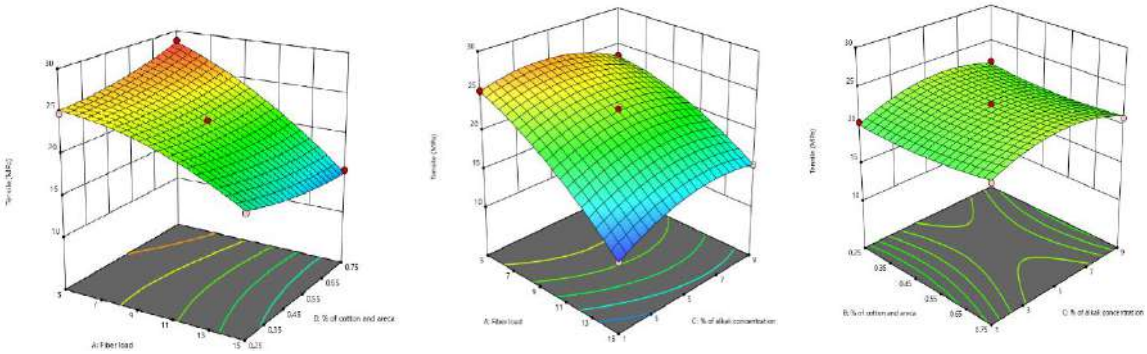
The ANOVA table for tensile strength shows the significant input parameters as well as significant corelating parameter and their square terms. Among the three varying parameters, fiber loading (F_L) and alkali concentration ($A_{\%}$) are the most significant. Among the interacting terms, interaction between fiber load and percentage of cotton and areca as well as interaction between fiber load and concentration of alkali, both were significant for the model. All the square terms were significant as well. The highest F value for fiber loading make it the most significant factor for the model. Table 5.2 shows the regression coefficient result for tensile strength (T_S)

Table: 5.2 Regression coefficients of RSM models for tensile strength (T_S)

Models	R-square	R-square (adjusted)	R-square(predicted)
T_S	0.997	0.9915	0.9596

R^2value ranges from 0 to 1. R=0.9985, then R^2=0.997, indicating that the linear connection between experimental values and network projected values can account for 99.7% of the entire variation in network prediction. The remaining 0.3% of the overall variance in network prediction is yet unknown. In this model for tensile strength, R^2 coefficients indicate that the model's prediction abilities are generally extremely good. Fig 5.2

illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on tensile strength (T_S).



(a) (b) (c)

Fig: 5.2 Surface plot of tensile strength

Table 5.3 shows the ANOVA table for flexural strength (F_S)

Table: 5.3 ANOVA for flexural strength (F_S)

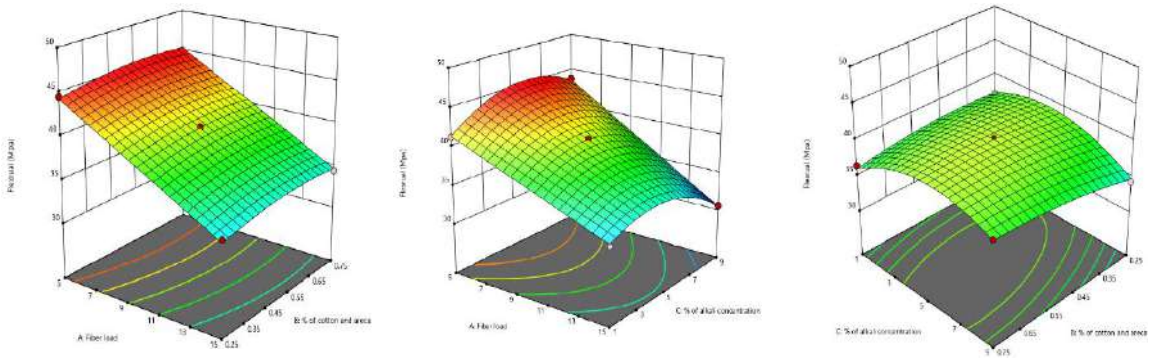
Source	Sum of squares	df	Mean square	F-value	p-value	Remarks
Model	271.07	9	30.12	161.62	< 0.0001	significant
F_L	223.34	1	223.34	1198.49	< 0.0001	significant
P_L	1.82	1	1.82	9.79	0.026	significant
$A_{\%}$	0.2701	1	0.2701	1.45	0.2825	not significant
$F_L \times P_L$	0.0064	1	0.0064	0.0343	0.8603	not significant
$F_L \times A_{\%}$	6.68	1	6.68	35.86	0.0019	significant
$P_L \times A_{\%}$	5.06	1	5.06	27.17	0.0034	significant
F_L^2	0.0025	1	0.0025	0.0137	0.9115	not significant
P_L^2	1.85	1	1.85	9.95	0.0252	significant
$A_{\%}^2$	32.82	1	32.82	176.1	< 0.0001	significant
Residual	0.9318	5	0.1864			
Lack of Fit	0.8454	3	0.2818	6.52	0.1358	not significant
Pure Error	0.0864	2	0.0432			
Total	272	14				

Fiber load (F_L), cotton areca percentage (P_L), interaction between F_L and $A_{\%}$, interaction between P_L and $A_{\%}$ and square of P_L and $A_{\%}$ were the significant factors for modeling of flexural strength. All of these factors p value is less than 0.05. The Model F-value of 161.62 implies the model is significant. Table 5.4 shows the regression coefficient result for flexural strength.

Table: 5.4 Regression coefficients of RSM models for flexural strength (F_S)

Models	R-square	R-square (adjusted)	R-square (predicted)
F_S	0.9966	0.9904	0.9496

In this model for flexural strength, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.3 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on flexural strength (F_S)



(a) (b) (c)

Fig 5.3 Surface plot of flexural strength

Table 5.5 shows the ANOVA table for impact strength (I_S)

Table 5.5 ANOVA for impact strength (I_S)

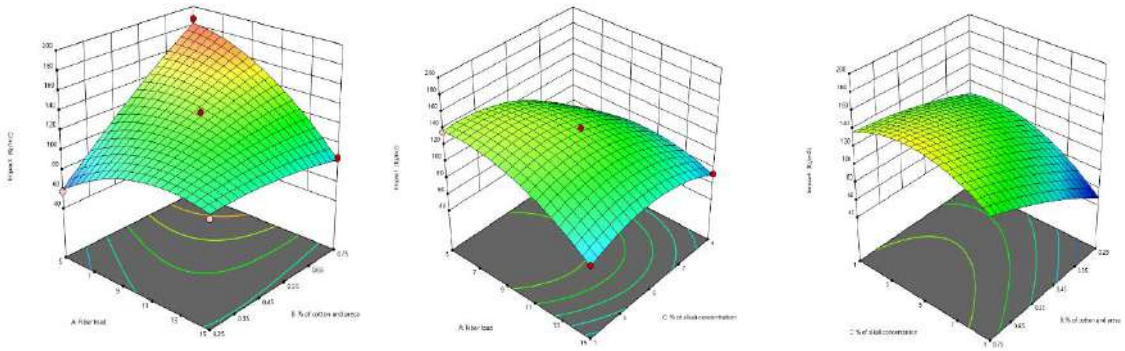
Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	19603.27	9	2178.14	90.84	< 0.0001	significant
F_L	1734.02	1	1734.02	72.32	0.0004	significant
P_L	5765.77	1	5765.77	240.47	< 0.0001	significant
$A_{\%}$	2675.83	1	2675.83	111.6	0.0001	significant
$F_L \times P_L$	4727.25	1	4727.25	197.16	< 0.0001	significant
$F_L \times A_{\%}$	1044.26	1	1044.26	43.55	0.0012	significant
$P_L \times A_{\%}$	432.22	1	432.22	18.03	0.0081	significant
F_L^2	1538.98	1	1538.98	64.18	0.0005	significant
P_L^2	110.81	1	110.81	4.62	0.0843	not significant
$A_{\%}^2$	1908.2	1	1908.2	79.58	0.0003	significant
Residual	119.89	5	23.98			
Lack of Fit	93.76	3	31.25	2.39	0.3084	not significant
Pure Error	26.13	2	13.06			
Total	19723.15	14				

All the factors are significant except the square of percentage of cotton and areca for modeling of impact strength. The model F-value of 90.84 implies the model is significant. Table5.6 shows the regression coefficient result for impact strength (I_S)

Table: 5.6 Regression coefficients of RSM models for impact strength (I_S)

Models	R-square	R-square (adjusted)	R-square (predicted)
I_S	0.9939	0.9830	0.9210

In this model for Impact strength, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.4 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration (A%) on impact strength (I_S)



(a) (b) (c)

Fig: 5.4 Surface plot of impact strength

Table 5.7 shows ANOVA for hardness (H)

Table: 5.7 ANOVA table for hardness (H)

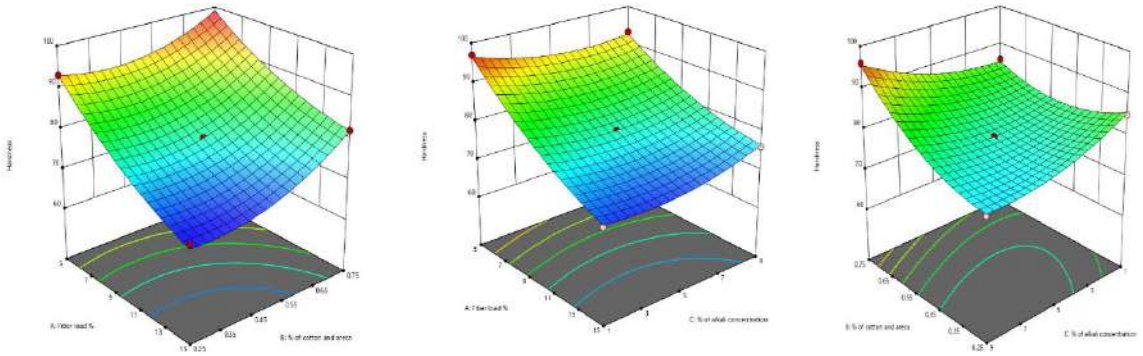
Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	1244	9	138.22	172.78	< 0.0001	significant
F_L	840.5	1	840.5	1050.62	< 0.0001	significant
P_L	180.5	1	180.5	225.62	< 0.0001	significant
$A_{\%}$	0.5	1	0.5	0.625	0.465	not significant
$F_L \times P_L$	12.25	1	12.25	15.31	0.0113	significant
$F_L \times A_{\%}$	20.25	1	20.25	25.31	0.004	significant
$P_L \times A_{\%}$	90.25	1	90.25	112.81	0.0001	significant
F_L^2	25.44	1	25.44	31.8	0.0024	significant
P_L^2	62.83	1	62.83	78.53	0.0003	significant
$A_{\%}^2$	25.44	1	25.44	31.8	0.0024	significant
Residual	4	5	0.8			
Lack of Fit	4	3	1.33			
Pure Error	0	2	0			
Total	1248	14				

All the factors are significant except concentration of alkali for modeling of hardness. The model F values of 172.78 implies the model is significant. Table 5.8 shows the regression coefficient result for hardness (H).

Table: 5.8 Regression coefficients of RSM models for H

Models	R-square	R-square (adjusted)	R-square (predicted)
H	0.9968	0.9910	0.9487

In this model for Impact strength, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.5 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on Hardness (H)



(a) (b) (c)

Fig 5.5 Surface plot of hardness

Table 5.9 shows ANOVA for moisture absorption (M_A)

Table: 5.9 ANOVA table for moisture absorption (M_A)

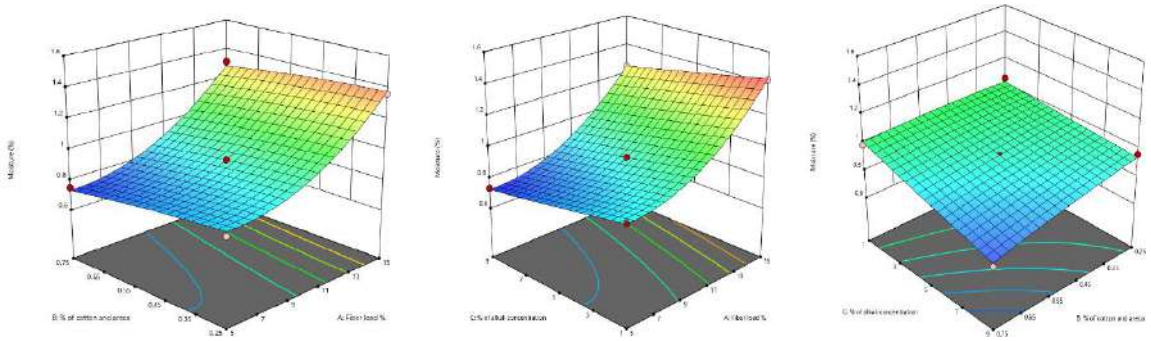
Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	0.741	9	0.0823	97.56	< 0.0001	significant
F_L	0.5698	1	0.5698	675.13	< 0.0001	significant
P_L	0.0252	1	0.0252	29.86	0.0028	significant
$A_{\%}$	0.0637	1	0.0637	75.51	0.0003	significant
$F_L \times P_L$	0.0003	1	0.0003	0.3629	0.5732	not significant
$F_L \times A_{\%}$	0	1	0	0.0296	0.8701	not significant
$P_L \times A_{\%}$	0.0086	1	0.0086	10.25	0.024	significant
F_L^2	0.0715	1	0.0715	84.68	0.0003	significant
P_L^2	0.0004	1	0.0004	0.5174	0.5042	not significant
$A_{\%}^2$	0.0001	1	0.0001	0.0657	0.8079	not significant
Residual	0.0042	5	0.0008			
Lack of Fit	0.0042	3	0.0014			
Pure Error	0	2	0			
Total	0.7452	14				

Fiber loading % (F_L), cotton and areca %, (P_L), concentration of alkali ($A_{\%}$), interaction between P_L and $A_{\%}$ and the square of F_L, are the significant factors for the model of moisture absorption. The model F-value of 97.56 implies the model is significant. Table 5.10 shows the regression coefficient result for moisture absorption (M_A).

Table: 5.10 Regression coefficients of RSM models for moisture absorption (M_A)

Models	R-square	R-square (adjusted)	R-square (predicted)
M_A	0.9943	0.9841	0.9094

In this model for moisture absorption, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.6 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on moisture absorption (M_A)



(a) **(b)** **(c)**

Fig 5.6 Surface plot of moisture absorption

Table 5.11 shows ANOVA for tensile modulus (T_M)

Table 5.11 ANOVA for tensile modulus (T_M)

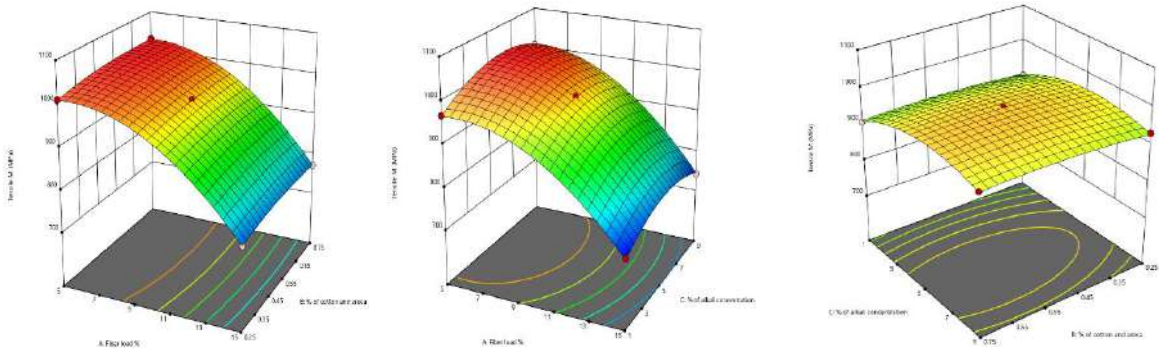
Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	**Remarks**
Model	1.51E+05	9	16764.48	1055.48	< 0.0001	significant
F_L	1.26E+05	1	1.26E+05	7948.78	< 0.0001	significant
P_L	325.13	1	325.13	20.47	0.0063	significant
$A_{\%}$	1800	1	1800	113.33	0.0001	significant
$F_L \times P_L$	36	1	36	2.27	0.1925	not significant
$F_L \times A_{\%}$	182.25	1	182.25	11.47	0.0195	significant
$P_L \times A_{\%}$	12.25	1	12.25	0.7712	0.42	not significant
F_L^2	17409.64	1	17409.64	1096.09	< 0.0001	significant
P_L^2	345.03	1	345.03	21.72	0.0055	significant
$A_{\%}^2$	6333.56	1	6333.56	398.76	< 0.0001	significant
Residual	79.42	5	15.88			
Lack of Fit	66.75	3	22.25	3.51	0.2294	not significant
Pure Error	12.67	2	6.33			
Total	1.51E+05	14				

All the factors are significant except the interaction between F_L and P_L as well as P_L and $A_{\%}$. The model F-value of 1055.48 implies the model is significant. Table 5.12 shows the regression coefficient result for tensile modulus (T_M)

Table: 5.12 Regression coefficients of RSM models for tensile modulus (T_M)

Models	R-square	R-square (adjusted)	R-square (predicted)
T_M	0.9995	0.9985	0.9927

In this model for tensile modulus, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.7 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on tensile modulus (T_M)



(a) (b) (c)

Fig 5.7 Surface plot of tensile modulus

Table 5.13 shows ANOVA for flexural modulus (F_M)

Table 5.13 ANOVA for flexural modulus (F_M)

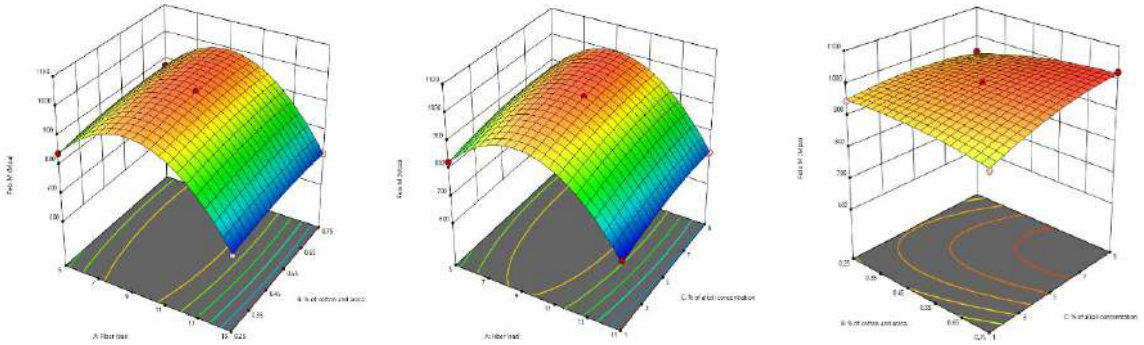
Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	**Remarks**
Model	1.90E+05	9	21058.31	318.87	< 0.0001	significant
F_L	42340.5	1	42340.5	641.12	< 0.0001	significant
P_L	932.47	1	932.47	14.12	0.0132	significant
$A_{\%}$	4033.37	1	4033.37	61.07	0.0005	significant
$F_L \times P_L$	110.25	1	110.25	1.67	0.2528	
$F_L \times A_{\%}$	30.25	1	30.25	0.458	0.5286	
$P_L \times A_{\%}$	2775.71	1	2775.71	42.03	0.0013	significant
F_L^2	1.39E+05	1	1.39E+05	2107.72	< 0.0001	significant
P_L^2	803.81	1	803.81	12.17	0.0175	significant
$A_{\%}^2$	1514.76	1	1514.76	22.94	0.0049	significant
Residual	330.21	5	66.04			
Lack of Fit	305.54	3	101.85	8.26	0.1099	not significant
Pure Error	24.67	2	12.33			
Total	1.90E+05	14				

All the factors are significant except the interaction between F_L and P_L as well as F_L and $A_{\%}$. The model F-value of 318.87 implies the model is significant. Table 5.14 shows the regression coefficient result for flexural modulus (F_M)

Table: 5.14 Regression coefficients of RSM models for flexural modulus (F_M)

Models	R-square	R-square (adjusted)	R-square (predicted)
T_M	0.9983	0.9951	0.9740

In this model for flexural modulus, R^2 coefficients indicate that the model's prediction abilities are extremely good. Fig 5.8 illustrates effect of fiber loading (F_L), percentage of cotton and areca (P_L) and alkali concentration ($A_{\%}$) on flexural modulus (F_M)



(a) **(b)** **(c)**

Fig 5.8 Surface plot of flexural modulus

5.2 Multi Objective Optimization with Desirability Function

Utilizing the response surface equations of the mechanical and physical responses, numerical optimization by desirability function is carried out. An objective function (D) called the desirability function has a value (di) between 0 and 1, with 1 being the greatest desirably. The function has the capacity to find a point in the specified design space within the constrained levels of factor settings and taking weight and importance into consideration that not only satisfy all the goals addressed as shown in tables 5.15 but also search for the most desirable value possible, di = 1.When a response is of the nominal-the-best (NTB) type, the response variable has both lower and upper specification limitations; as a result, the desirability function has a value of 0 when the response variable

hits one of these limits. In the case of a smaller-the-better (STB) type of response (or a larger-the-better [LTB] type of response), the response variable only has an upper (or lower) specification limit; as a result, the desirability function has a value of 0 when the response variable reaches the upper (or lower) specification limit. Following the definition of the desirability functions for each individual response variable, these functions are combined into a single measure known as the overall desirability function. The general desirability function may be calculated using the geometric mean or weighted geometric mean of each desire function [Lee et al. 2018]. The goal of this optimization procedure is to arrive at the optimal values of factor settings that provide the highest tensile strength, flexural strength, impact strength, hardness, tensile modulus, and flexural modulus, as well as the lowest moisture absorption. The desirability function is given by eqn. (5.10), where n is the total number of replies. A goal's weight value can be changed to change the specific desirability function's form. All of the variables and responses in this case have equal weight values given to them.

The factors are given a 3 out of 5 importance rating and weight of 1 for a neutral setting.

$$D = (d_1 x d_2\, x \dots \dots . x d_n)^{\frac{1}{n}} = (\prod_{i=1}^{n} d_i)^{\frac{1}{n}} \quad (5.10)$$

For goal as a target, the desirability can be defined as following formula.

$d_i = 0,$ $\quad \text{Response}_i \leq \text{Low}_i$

$d_i = \left\lfloor \frac{Response_i - Low_i}{Target_i - Low_i} \right\rfloor^{wt_i},$ $\quad \text{Low}_i < \text{Response}_i < \text{Target}_i$

$d_i = 1,$ $\quad \text{Response}_i = \text{Target}_i$

$d_i = \left\lfloor \frac{High_i - Response_i}{High_i - Target_i} \right\rfloor^{wt_i},$ $\quad \text{Target}_i < \text{Response}_i < \text{High}_i$

$d_i = 0,$ $\quad \text{Response}_i \geq \text{Low}_i$

The following formals will determine desirability for a goal within a range (a constraint).

$d_i = 0,$ $\quad \text{Response}_i \leq \text{Low}_i$

$d_i = 1,$ $\quad \text{Low}_i < \text{Response}_i < \text{High}_i$

$d_i = 0,$ $\quad \text{Response}_i \geq \text{High}_i$

Table 5.15 shows the constraint for multi objective optimization.

Table: 5.15 Constraint for multi objective optimization

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
Fiber load	is in range	5	15	1	1	3
% of cotton and areca	is in range	0.25	0.75	1	1	3
% of alkali concentration	is in range	1	9	1	1	3
Tensile strength	maximize	12.16	28.75	1	1	3
Flexural strength	maximize	30.54	44.95	1	1	3
Impact	maximize	51.42	186.4	1	1	3
Hardness	is target = 98	70	98	1	1	3
Moisture absorption	minimize	0.712	1.43	1	1	3
Tensile modulus	maximize	732	1030	1	1	3
Flexural modulus	maximize	694	1021	1	1	3

The optimum combination for making hybrid cotton-areca reinforced polypropylene composite is stated in table 5.16. The combination with the highest desirability (=0.927) was found for 5.667% total fiber load, 5.869% of alkali concentration and cotton and areca percentage in the fiber load respectively 75% and 25% which yielded tensile strength= 27.664MPa, flexural strength= 44.9MPa, impact strength =174.623KJ/m^2, hardness=98, moisture absorption= 0.705%, tensile modulus= 1031.895MPa and flexural modulus= 920.634Mpa. In Fig. 5.9 bar chart of desirability analysis with individual desirability and overall desirability is depicted.

Table:5.16 Desirability optimization solution

Number	Fiber load	% of cotton and areca	% of alkali concentration	Tensile strength	Flexural strength	Impact strength	Hardness	Moisture absorption	Tensile modulus	Flexural modulus	Desirability	
1	**5.667**	**0.750**	**5.869**	**27.664**	**44.900**	**174.623**	**98.000**	**0.705**	**1031.895**	**920.634**	**0.927**	**Selected**
2	5.652	0.750	5.832	27.693	44.909	174.942	97.999	0.706	1031.882	919.487	0.927	
3	5.680	0.750	5.899	27.639	44.891	174.360	97.998	0.704	1031.897	921.629	0.927	
4	5.697	0.750	5.942	27.604	44.879	173.975	98.000	0.703	1031.894	922.980	0.927	
5	5.628	0.750	5.775	27.738	44.923	175.435	98.000	0.708	1031.844	917.643	0.927	
6	5.667	0.750	5.792	27.699	44.884	175.222	97.920	0.707	1031.792	919.832	0.927	
7	5.716	0.750	5.869	27.624	44.845	174.516	97.876	0.705	1031.779	923.186	0.927	
8	5.629	0.750	5.727	27.759	44.911	175.803	97.950	0.709	1031.758	917.135	0.927	

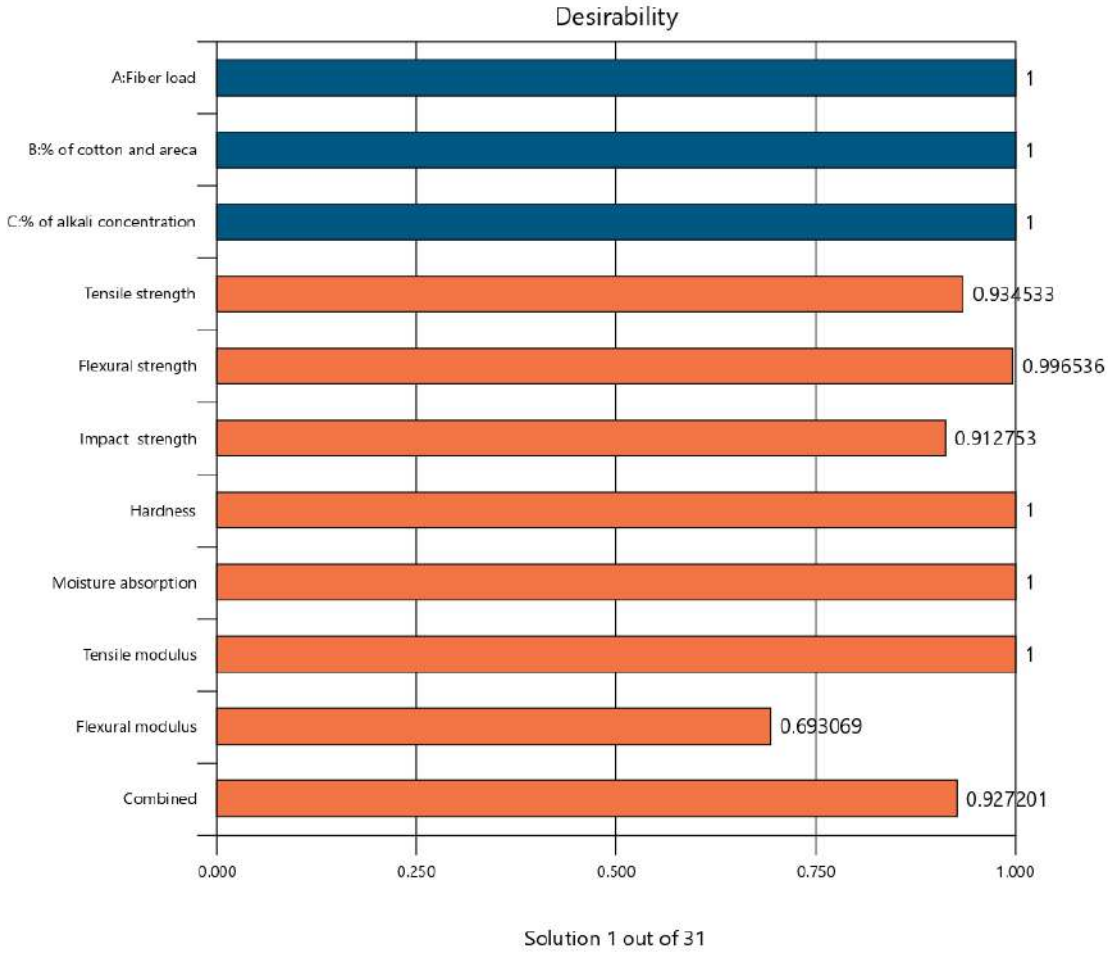


Fig: 5.9 Desirability analysis of optimum composite combination

Chapter-6

Discussion on Results

6.1 Mechanical Properties

There are many mechanical properties for a natural fiber reinforced composite that is needed to be characterized for determining their applications in different sphere of life. In the present study tensile strength, tensile modulus, flexural strength, flexural modulus, impact strength and hardness are observed from experimental run as well as prediction derived from RSM model. From previous scientific research it is evident that the aforementioned mechanical properties are the mostly used properties among all the other ones.

A material's tensile strength is the highest stress it can endure in tension before breaking. The composite's load-bearing capability and structural integrity may both be evaluated using this crucial attribute. In situations where tensile loads are a concern, determining the tensile strength aids in ensuring the structural integrity of fiber reinforced polymer (FRP) composites. Tensile strength and FRP composites' ability to support loads are closely correlated. It establishes the maximum tensile load that the composite can withstand before failing. Engineers can design FRP structures and components that can handle the projected mechanical loads by understanding the tensile strength. Engineers may verify a material's appropriateness for certain applications and guarantee that it can safely handle the anticipated tensile loads without catastrophic failure by analyzing the material's tensile strength. Engineers may choose the right fiber types, fiber volume fractions, and matrix materials to accomplish required strength requirements while minimizing weight and cost by taking the material's tensile strength into account. In present study tensile strength of novel cotton areca fiber reinforced polypropylene composite has been observed. Previous research has shown that factors such as fiber

loading, fiber type, alkali treatment type, alkali treatment time, hybridization, manufacturing technique, matrix material, and fiber length may all affect tensile strength. The hybrid composite's tensile behavior is characterized by figure 4.7, where the maximum tensile strength of 28.75 MPa was discovered for 5% fiber load, 5% NaOH concentration, and 75% cotton and 25% areca fiber percentage (Experiment run 9). The tensile strength of the composite without alkali treatment was 19.9 MPa, which is 31% less than the maximum value. As a result of the untreated fiber composite having the same fiber load (5%) and volume fraction of cotton (75%) and areca (25%) as the treated fiber composite, the effect of alkali treatment is clearly visible.

However, it is challenging to generalize about the others given the different input factors. By examining the tensile strength ANOVA table and identifying the major variables influencing tensile strength, this confusion was resolved. The two most important variables among the three are fiber loading and alkali content, according to table 5.1. A few interacting factors and square terms have an impact on tensile strength as well. The RSM model of tensile strength prioritizes fiber loading as the most important element since it has the highest F value. The model accuracy was also shown by comparing experimental tensile strength values with predicted values from RSM model in table 6.1. By dividing the subtracted value of the predicted and experimental value by experimental value and then multiplying the result by 100, the mean absolute percent of error was derived. The predicted model is also supported by the mean absolute percent of error (MAPE), which was 0.97%. Fig 6.1 shows a comparison between Experimental value and predicted value of tensile strength.

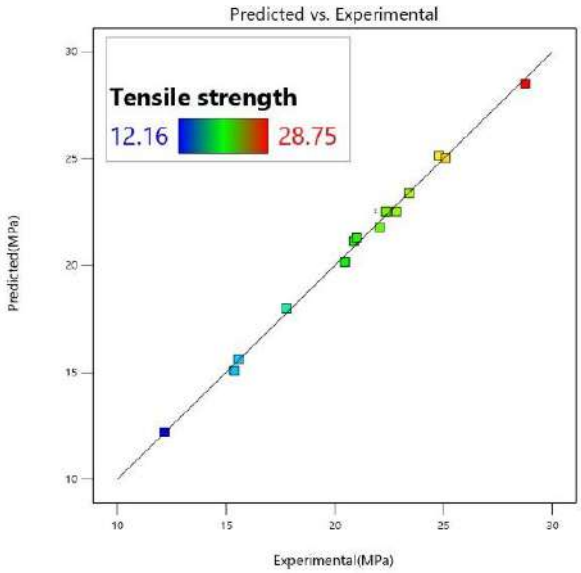


Fig: 6.1 Tensile strength experimental and predicted value comparison

Table: 6.1 Experimental versus predicted value of tensile strength (T_S)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Tensile strength		APE %
				Experimental	Predicted	
1	10	75+25	9	21.01	21.28	1.30
2	15	25+75	5	17.77	18.00	1.27
3	10	25+75	9	22.07	21.79	1.25
4	15	50+50	9	15.58	15.63	0.32
5	5	50+50	1	25.1	25.05	0.20
6	5	25+75	5	24.81	25.13	1.31
7	10	50+50	5	22.36	22.52	0.70
8	10	25+75	1	20.45	20.18	1.34
9	5	75+25	5	28.75	28.52	0.79
10	10	50+50	5	22.83	22.52	1.37
11	5	50+50	9	23.43	23.38	0.20
12	10	50+50	5	22.36	22.52	0.70
13	10	75+25	1	20.87	21.15	1.32
14	15	75+25	5	15.39	15.07	2.10
15	15	50+50	1	12.16	12.21	0.39

An object's capacity to sustain bending or flexural stresses without breaking or malfunctioning is referred to as its flexural strength when made of a natural fiber reinforced polymer composite. It serves as a gauge for the highest load or stress that a composite material can withstand while bending without cracking. The performance and behavior of natural fiber reinforced polymer composites under bending circumstances may be predicted using flexural strength as a foundation. It assists in making estimates for

things like the composite's maximum permissible bending stress, deflection, and overall durability, enabling trusted and secure use in real-world scenarios. Present study observed flexural strength of 16 composites which was fabricated using RSM design. According to prior studies, the volume fraction of various fibers in fiber loading, fiber orientation, type of polymer matrix, various fiber treatments, fabrication method, moisture content, and environmental conditions all affect flexural strength in a fiber reinforced composite. In this study, we tested the volume fraction of cotton and areca fibers, the percentage of alkali concentration, and the amount of fiber loading on the flexural strength using both experimental methods and data from an ANOVA table, which clearly demonstrates the importance of the aforementioned parameters as well as their interacting terms and square terms. Figure 4.7 shows that the maximum value of flexural strength, 44.95 MPa, was obtained for 5% fiber load, 5% NaOH concentration, and 75% cotton and 25% areca fiber percentages (Experiment run 9). With the same fiber load and volume percent of cotton and areca, an untreated composite had a flexural strength of 37.98 MPa, which is 15.51% less than the maximum result. It unmistakably demonstrates how alkali treatment of fibers affects the properties.

We know from prior research that alkali treatment cleans the fiber surface of undesired, weak, non-load-bearing components including lignin, pectin, hemicellulose, and other waxy impurities, leaving behind a more pure, chemically active fiber that is more compatible with the matrix. Table 5.3 shows that the volume fraction of cotton and areca as well as the fiber loading have a substantial impact on the flexural strength model. The fiber loading has the highest F value, making it the most important factor. A few interacting factors and square terms have an impact on tensile strength as well. Comparing experimental and RSM model predictions for flexural strength in Table 6.2 further demonstrated the model’s correctness. Fig 6.2 shows comparison between experimental value and predicted value. The predicted value is also validated by the MAPE which is only 0.59%

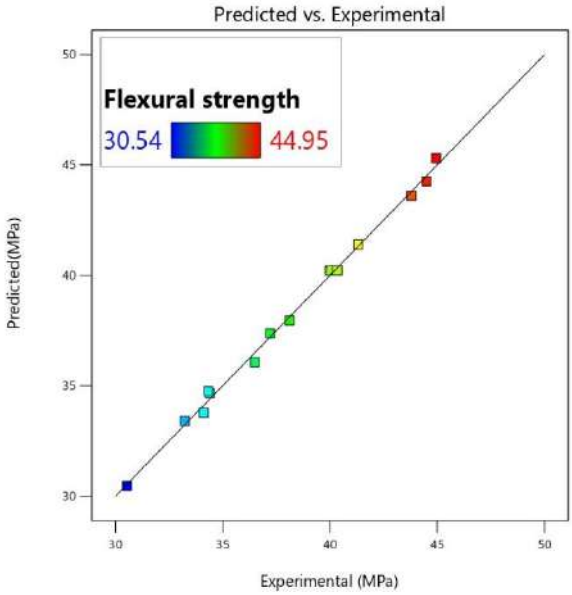


Fig: 6.2 Experimental and predicted value comparison of flexural strength

Table: 6.2 Experimental versus predicted value of flexural strength (F_S)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Flexural		APE %
				Experimental	Predicted	
1	10	75+25	9	38.12	37.96	0.42
2	15	25+75	5	34.12	33.77	1.01
3	10	25+75	9	34.33	34.75	1.23
4	15	50+50	9	30.54	30.46	0.25
5	5	50+50	1	41.32	41.40	0.19
6	5	25+75	5	44.5	44.26	0.54
7	10	50+50	5	40.35	40.23	0.30
8	10	25+75	1	37.21	37.37	0.43
9	5	75+25	5	44.95	45.30	0.77
10	10	50+50	5	39.99	40.23	0.60
11	5	50+50	9	43.8	43.62	0.42
12	10	50+50	5	40.35	40.23	0.30
13	10	75+25	1	36.5	36.08	1.16
14	15	75+25	5	34.41	34.65	0.69
15	15	50+50	1	33.23	33.42	0.56

The capacity of natural fiber reinforced polymer composites to sustain rapid or dynamic loading without cracking is measured by their impact strength, a crucial mechanical attribute. It shows how resilient a material is against pressures from contact or impact. The impact strength is influenced by variables like fiber type, fiber volume

percentage, fiber orientation, matrix characteristics, and interfacial adhesion. Improving impact resistance requires proper fiber dispersion, alignment, and bonding inside the composite. A part is also played by the production procedure, which includes the cure environment and post-processing steps. For situations where the composite is subjected to abrupt impacts or collisions, maximizing impact strength is essential for maintaining dependability and performance. In the present study effect of fiber loading, volume fraction of cotton and areca fibers and percentage of alkali concentration is observed on impact strength. The highest impact strength measurement was 186.4 KJ/m^2, exceeding the impact strengths of most natural fibers and, in certain circumstances, synthetic fibers like glass and nylon reinforced polymer composites [Kusaseh et al. 2018]. The maximum result is shown in Experiment Run 9 with 5% fiber load, 5% NaOH concentration, 75% cotton, and 25% areca fiber percentages. The untreated composite with the same volume fractions and fiber load displays 100.3 KJ/m^2, which is 46.2% less than the highest value. This phenomenon explains the impact of alkali treatment on impact strength. From ANOVA table 5.5 we can see the that all the aforementioned factors are significant due to high value of F. Volume fraction of cotton and areca fiber seem to be the most significant for impact strength with highest F value. Fig 6.3 shows comparison of predicted and experimental values and table 6.3 provides validation for the model by calculating MAPE as 2.63%

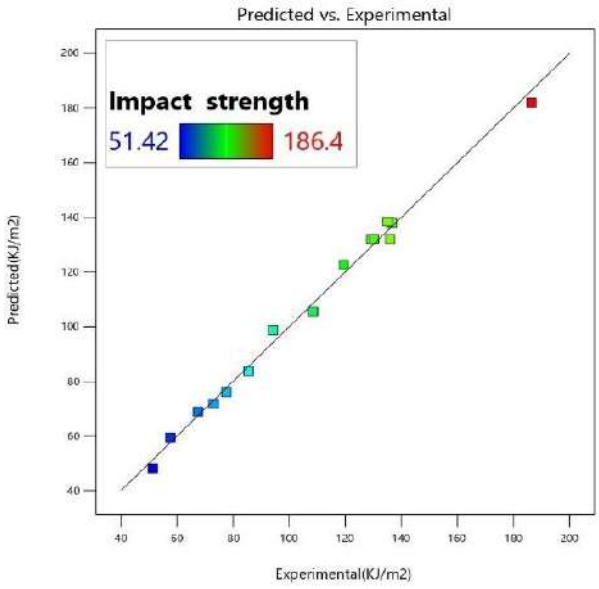


Fig: 6.3 Experimental and predicted value comparison of impact strength

Table: 6.3 Experimental versus predicted value of impact strength (I_S)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Impact strength		APE %
				Experimental	Predicted	
1	10	75+25	9	119.6	122.61	2.52
2	15	25+75	5	94.3	98.78	4.75
3	10	25+75	9	51.42	48.13	6.41
4	15	50+50	9	73.05	71.86	1.62
5	5	50+50	1	136.7	137.89	0.87
6	5	25+75	5	57.65	59.47	3.16
7	10	50+50	5	136	131.87	3.04
8	10	25+75	1	108.5	105.49	2.77
9	5	75+25	5	186.4	181.92	2.40
10	10	50+50	5	129.3	131.87	1.99
11	5	50+50	9	67.52	68.99	2.18
12	10	50+50	5	130.3	131.87	1.20
13	10	75+25	1	135.1	138.40	2.44
14	15	75+25	5	85.54	83.72	2.13
15	15	50+50	1	77.6	76.13	1.90

Hardness is a crucial mechanical characteristic that describes how resistant a natural fiber reinforced polymer composite is to being scratched or indented. It gauges a material's resistance to surface buckling or penetration by outside pressures. The kind of natural fiber employed, the composition and structure of the polymer matrix, and the interfacial adhesion between the fibers and the matrix are all variables that affect the hardness of natural fiber reinforced polymer composites. Increased hardness can result from consistent fiber dispersion and proper fiber-matrix bonding. For applications where resistance to wear, abrasion, or surface damage is essential, understanding and optimizing hardness is crucial for assuring the composite's endurance and lifetime.

In the present study effects of fiber load, volume fraction of cotton and areca and percentage of alkali concentration was observed on hardness. Neat polypropylene’s hardness is near 90-92. And our highest value of hardness was 99, which definitely shows some improvement due to inclusion of natural fibers. Hardness of untreated composite was 84, which was 15.15% less than the highest value. It shows the effect of alkali treatment on composite. But from the ANOVA of table 5.7 finds the fiber load and volume fraction of cotton and areca fibers more significant as well as some interacting terms like interaction between fiber load and volume fraction, fiber load and alkali concentration and volume

fraction and alkali concentration as well as square terms of all the input parameters. In fig 5.1 normality plot of hardness does not show enough normality like the other properties which can be explained by research of Schmidt and Finan [2018] which mentions that the findings are frequently not significantly affected by breaches of this normalcy assumption in high sample sizes (for example, if there are more than ten observations for each variable, in our case 15). Figure 6.4 illustrates the similarity between experimental and anticipated hardness values, and the computation of MAPE illustrates the validity of the RSM model for hardness. MAPE in this instance was 0.48%. Table 6.4 provides the calculations.

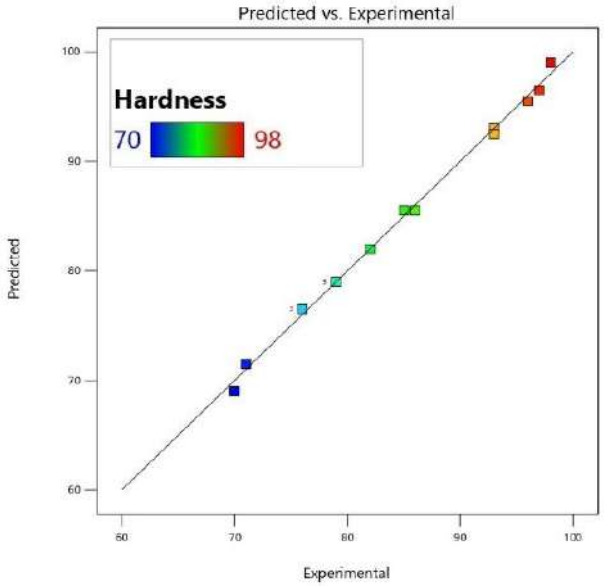


Fig: 6.4 Experimental and predicted value comparison of Hardness

Table: 6.4 Experimental versus predicted value of hardness (H)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Hardness		APE %
				Experimental	Predicted	
1	10	75+25	9	96	95.50	0.52
2	15	25+75	5	70	69.00	1.43
3	10	25+75	9	76	76.50	0.66
4	15	50+50	9	76	76.50	0.66
5	5	50+50	1	97	96.50	0.52
6	5	25+75	5	93	93.00	0.00
7	10	50+50	5	79	79.00	0.00
8	10	25+75	1	85	85.50	0.59
9	5	75+25	5	98	99.00	1.02
10	10	50+50	5	79	79.00	0.00
11	5	50+50	9	93	92.50	0.54
12	10	50+50	5	79	79.00	0.00
13	10	75+25	1	86	85.50	0.58
14	15	75+25	5	82	82.00	0.00
15	15	50+50	1	71	71.50	0.70

An essential mechanical characteristic that describes the stiffness or rigidity of a natural fiber reinforced polymer composite is called the tensile modulus. It gauges how easily a material will bend in the presence of tensile or stretching forces. The kind of natural fiber utilized, fiber volume fraction, fiber orientation, and the characteristics of the polymer matrix are only a few examples of the variables that might affect the tensile modulus in natural fiber reinforced polymer composites. Higher tensile modulus values are often the consequence of increasing the fiber content and aligning the fibers in the loading direction. Tensile modulus, which evaluates whether the composite can endure elongation without permanently deforming, is crucial for situations where structural stability and dimensional stability are crucial.

The highest result, found for experiment run 9 with 5% fiber load, 75% volume fraction of cotton, 25% of areca, and 5% of alkali concentration, was 1030MPa in the current study's analysis of the tensile modulus values of 16 composites with various input parameters. We found that the tensile modulus for the untreated fiber was 910MPa. This is 7.76% behind the highest possible number. The concentration of alkali concentration used to treat the fiber was the sole difference between the two composites, and it is obvious that

the elimination of undesired impurities results in higher attributes for the composite treated with alkali. We may infer the significant effects of the aforementioned input factors, such as fiber loading, cotton and areca volume fractions, and alkali %, using ANOVA table 5.11. All three of them are substantial, as seen by the high F value, with fiber loading having the greatest F value. Additionally, it demonstrates the interaction between several of these factors and the significance of their p value and F value. The only substantial interaction, for example, was discovered to exist between fiber load and alkali content. On the other hand, all of the input parameters' square terms appear to be significant. From fig 4.7 we can see the tensile modulus value of all 15 composite, and from fig 6.5 we can see the comparison between experimental value and predicted value. We can also validate our RSM model for tensile modulus by calculating MAPE which is shown on table 6.5 with a value of 0.23%.

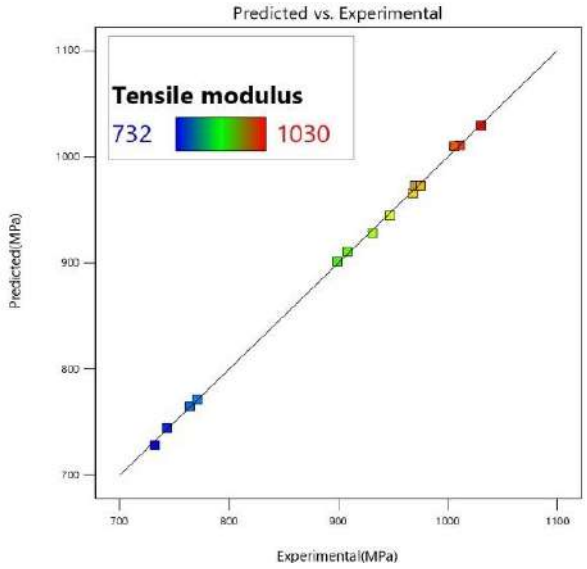


Fig: 6.5 Experimental and predicted value comparison of tensile modulus

Table: 6.5 Experimental versus predicted value of tensile modulus (T_M)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Tensile modulus		APE %
				Experimental	Predicted	
1	10	75+25	9	947	944.38	0.28
2	15	25+75	5	764	765.00	0.13
3	10	25+75	9	931	928.13	0.31
4	15	50+50	9	743	744.88	0.25
5	5	50+50	1	968	966.13	0.19
6	5	25+75	5	1011	1010.25	0.07
7	10	50+50	5	975	972.33	0.27
8	10	25+75	1	899	901.63	0.29
9	5	75+25	5	1030	1029.00	0.10
10	10	50+50	5	970	972.33	0.24
11	5	50+50	9	1006	1009.63	0.36
12	10	50+50	5	972	972.33	0.03
13	10	75+25	1	908	910.88	0.32
14	15	75+25	5	771	771.75	0.10
15	15	50+50	1	732	728.38	0.50

The resistance of natural fiber reinforced polymer composites to bending or flexural deformation is indicated by their flexural modulus. It describes how stiff the composite is under bending pressures. Natural fiber composites often have lower flexural modulus than composites reinforced with synthetic fibers. This is explained by the reduced stiffness of natural fibers by nature and the difficulties in generating strong interfacial bonding. To increase the flexural modulus of natural fiber composites, however, techniques including fiber surface treatment, finding optimal fiber loading and optimal level of volume fraction of different fibers, fiber alignment improvement, and matrix upgrades can be used. These methods seek to promote load transmission and boost fiber-matrix interaction, which will improve the composite material's flexural performance.

In the current investigation, experiment run 1 (with fiber load 10%, cotton 75%, areca 25%, and alkali concentration 9%) had the greatest flexural modulus, which was measured to be 1021 MPa. The composite that had not been treated displayed a value of 810MPa, 20.67% less than the maximum result. Experiment 9 provides us a value of 947MPa, which is 14.56% higher and clearly demonstrates the influence of alkali treatment on flexural modulus. It contains the identical combination as the untreated combination except from the alkali treatment element. We can see the variables of significance in ANOVA table 5.13. All of the aforementioned parameters appear to be significant in this situation, with fiber loading having the greatest F value and appearing to

be the most important. Interaction between volume fraction of cotton-areca and alkali concentration is significant, but the other two interacting terms are not significant enough. All the square term has significance according to F and p value. Fig 6.6 shows comparison between predicted and experimental runs. The RSM model validation is shown by Table 6.6 with a MAPE 0.93%

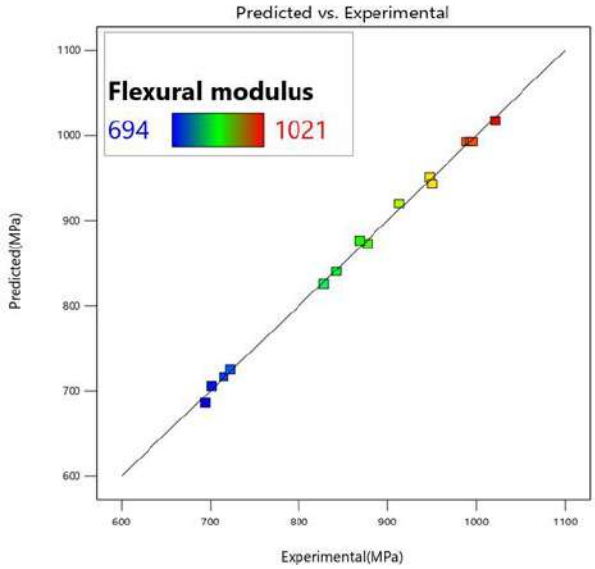


Fig: 6.6 Experimental and predicted value comparison of flexural modulus

Table: 6.6 Experimental versus predicted value of flexural modulus (F_M)

Experimental run	Fiber load %	Cotton areca %	NaOH %	Flexural modulus		APE %
				Experimental	Predicted	
1	10	75+25	9	1021	1017.25	0.37
2	15	25+75	5	714	705.45	1.20
3	10	25+75	9	949.63	942.97	0.70
4	15	50+50	9	708	725.20	2.43
5	5	50+50	1	828	825.80	0.27
6	5	25+75	5	842	840.45	0.18
7	10	50+50	5	996	992.67	0.33
8	10	25+75	1	947	950.75	0.40
9	5	75+25	5	877	872.55	0.51
10	10	50+50	5	989	992.67	0.37
11	5	50+50	9	868	876.20	0.95
12	10	50+50	5	993	992.67	0.03
13	10	75+25	1	913	919.66	0.73
14	15	75+25	5	694	716.55	3.25
15	15	50+50	1	701	685.80	2.17

6.2 Physical Properties

Natural fiber reinforced composites are significantly affected by moisture absorption. Natural fibers' capacity to hold onto moisture can have an impact on the composites' general performance and physical characteristics. Moisture absorption can eventually cause the composite structure to weaken, distort, and alter in dimensions. The fiber-matrix interface may deteriorate and the mechanical qualities may be diminished by chemical and physical deterioration, such as hydrolysis. For natural fiber reinforced composites to be long-lasting, dimensionally stable, and mechanically sound, especially in applications exposed to high humidity or wet environments; it is essential to understand and control moisture absorption. The performance and dependability of these composites must be maintained through the use of appropriate moisture protection and mitigation measures, including surface treatments, moisture barriers, and suitable fiber-matrix combinations.

In the current investigation, 16 composite specimens with various combinations of fiber load, cotton-areca volume fraction, and alkali concentration % were examined, and the moisture absorption rate was noted. In contrast to mechanical qualities, we don't need to enhance moisture intake %; rather, we need to reduce the rate, which will be advantageous to the composite in various circumstances. Figure 5.7 shows that the experiment run 1 with a 10% fiber load, cotton-areca (75%-25%) volume fraction, and 9% concentration of alkali treatment had the lowest moisture absorption rate of 0.712%. The composite that has not been treated has a moisture intake rate of 2.13%. With the same combination of fiber load and volume fraction as experiment run 9, with the exception of the 5% alkali treatment, the moisture absorption rate is comparatively low at 0.749%, which is 65% lower than untreated composite and demonstrates the impact of alkali treatment on fiber. By eliminating unwanted components, fibers become less hydrophilic and more hydrophobic, facilitating their usage in a variety of situations where they may come into contact with moisture or water. Fig 6.7 shows the comparison between experimental and predicted value and table 6.7 validates the RSM model of moisture absorption with a MAPE of 1.39%.

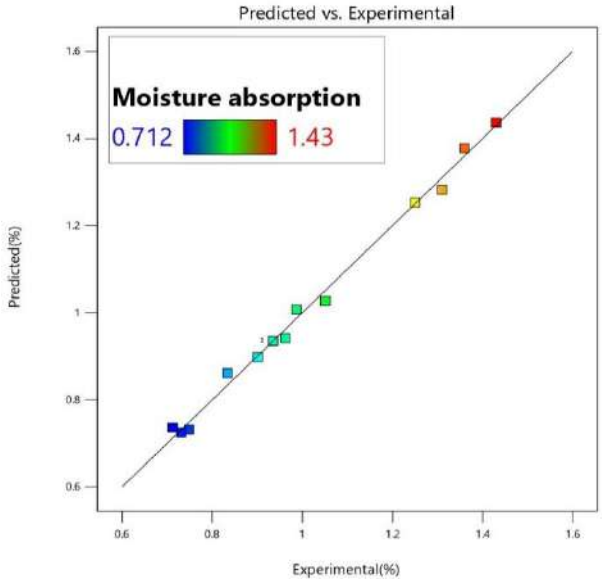


Fig: 6.7 Experimental and predicted value comparison of moisture absorption%

Table: 6.7	Experimental versus predicted value of moisture absorption (M_A)					
Experimental run	Fiber load %	Cotton areca %	NaOH %	Moisture absorption %		APE %
				Experimental	Predicted	
1	10	75+25	9	0.71	0.74	3.39
2	15	25+75	5	1.36	1.38	1.29
3	10	25+75	9	0.96	0.94	2.14
4	15	50+50	9	1.25	1.25	0.25
5	5	50+50	1	0.90	0.90	0.35
6	5	25+75	5	0.83	0.86	3.27
7	10	50+50	5	0.94	0.94	0.00
8	10	25+75	1	1.05	1.03	2.29
9	5	75+25	5	0.75	0.73	2.34
10	10	50+50	5	0.94	0.94	0.00
11	5	50+50	9	0.73	0.72	0.91
12	10	50+50	5	0.94	0.94	0.00
13	10	75+25	1	0.99	1.01	2.09
14	15	75+25	5	1.31	1.28	2.08
15	15	50+50	1	1.43	1.44	0.46

Natural fiber reinforced composites may be examined in detail at the microscale using scanning electron microscopy (SEM), a useful tool. The surface morphology of the composite may be examined using high-resolution pictures from a scanning electron microscope, which also enables the study of any damage or flaws. It allows for the

inspection of the microstructure of the composite, including the positioning and dispersion of the fibers inside the matrix. Debonding, fractures, and voids that could impair the composite's mechanical characteristics can be found through SEM examination.

In our present study we have examined 4 composite samples with no alkali treatment, 1% alkali treated, 5% alkali treated and 9% alkali treated. We can see from figure 4.10 the untreated composite had an abundance of microfibrils (lignin, hemicellulose, pectin, wax, and contaminants) and the fiber surface was not clear. These two processes promote poor fiber matrix bonding, load bearing capacity difficulties, increased moisture absorption, poor mechanical and physical qualities, and so forth. In section 6.1, we also discovered that untreated fiber composites had much worse mechanical characteristics than treated composites. The 1% treated composite shows slight improvement where the amount of microfibril seem less and the surface looks smoother than the untreated composite. The elimination of undesirable non-loadbearing components improved significantly in the 5% alkali treated composite. The fiber surface appears cleaner while surface roughness shows improvement, allowing for better adhesion between fiber and polymer matrix. We can also observe from the preceding section that almost every 5% alkali treated composites specially experiment run 9 had much improved mechanical characteristics than the rest of the fiber composites, with the exception of flexural modulus. The 9% alkali treated fiber composite displays no microfibrils and an improvement in surface roughness. However, the mechanical characteristics of 9% alkali treated fibers did not improve significantly in comparison to the 5% alkali treated composite. This occurrence implies that a larger concentration of alkali fraction has also removed part of the essential cellulose from the fibers, making them thinner and weaker, allowing for significantly lower mechanical characteristics of composites. Based on the SEM pictures and mechanical characteristics, we can conclude that the ideal concentration of alkali treatment for cotton-areca fiber reinforced polymer composite is around 5%. Our desirability function analysis for determining the ideal settings also discovered that the optimum alkali concentration is 5.869%, confirming our assertion.

From X-ray diffraction (XRD) graphs we have observed some distinct peaks for polypropylene mostly. It is vital to note that the presence of the polymer matrix can alter the XRD pattern when cotton and areca fibers are integrated into the matrix to produce a composite. Because the diffraction from the polymer might overlap with the diffraction from the cellulose, the peaks belonging to the cellulose may become less clear or changed due to the presence of the polymer matrix. As a result, while the cellulose I peak may still be present in a cotton fiber reinforced polymer composite, its intensity and form may be altered by interactions between the cotton fibers and the polymer matrix. From figure 4.9 we can see distinct peak at $2\theta = 12.54^{o}$, 14.01^{o}, 15.25^{o}, 15.97^{o}, 16.72^{o}, 18.38^{o}, 19.6^{o}, 21.64^{o}, 21.00^{o}, 21.64^{o}, 25.32^{o}, 28.19^{o}, 42.41^{o}. From previous research studies it is evident that 14.01^{o}, 16.72^{o}, 21.00^{o}, 21.64^{o}, 25.32^{o} are distinct peak of polypropylene [Ariyoshi et al. 2021] and 12.54^{o}, 15.97^{o}, 16.72^{o} are distinct peak for cellulose and hemicellulose, lignin and pectin [Escobaret al. 2022] The 16.72^{o} peak of cellulose, hemicellulose lignin and pectin is overlapped by similar peak of polypropylene. But it was observed in figure 4.9b, 4.9c, 4.9d and 4.9e that alkali treatments reduced its intensity in comparison with the untreated fiber composite shown in fig 4.9(a). By observing cellulose peak 12.54^{o} from figure 4.9 we also came to learn that, its intensity reaches its peak for 5% alkali treated composite. The lowest peak intensity was found for 1% treated composite. The 9% alkali treated composite shows slight decrease of the same peak in comparison with the highest intensity but still remaining higher than the untreated specimen. Which is the proof of more than the recommended amount of alkali treatment and the decrease of certain portion of cellulose along with other non-load bearing components. Similar phenomena to those seen with peak 16.72^{o} have also been seen with peak 21.64^{o}, where the peak intensity was larger for untreated composite and decreased in other alkali treated composite, demonstrating the effectiveness of alkali treatment in eliminating undesired contaminants.

The same selected composites' crystallinity indices are shown in Table 4.2, and it has been discovered that alkali treatment has boosted the composite's overall crystallinity. Untreated composite had the lowest crystallinity index, which was 64.73 % and the highest, which was 72.13% for specimen 5. The phenomenon is proof that alkali treatment increased crystallinity. The crystal size was also determined in the current study, with the untreated composite having a crystal size of 9.43 nm and the treated composite having a crystal size of 12 nm, which is 27.25% larger. Greater crystal size in the treated composite

compared to the untreated composite may indicate a decrease in hydrophilic nature [Bright et al. 2021]. The same result has been reached after looking at fig. 4.7 of moisture absorption characteristics. The composite that was alkali-treated at 1% and 5% showed almost identical crystal sizes at 10.18 nm. Polypropylene, cotton, and areca all have lower crystalline indices than 65% on their own. We have increased the proportion of crystallinity in every case of hybridization. Increased strength, improved thermal stability, decreased permeability, and a higher melting point, among other properties, can be attributed to greater crystallinity.

6.3 Chemical Properties

Fourier transform infrared spectroscopy, also known as FTIR, is a useful analytical method for describing composites made of natural fibers. It enables scientists to investigate the chemical structure and bonding of these materials. The FTIR technique measures the infrared radiation's absorption and sheds light on the functional groups in natural fibers and how they interact with the composite matrix.

From the photographic view of FTIR spectra in fig 4.8 shows the FTIR spectra of Experiment run 6(1% alkali treated), 5(5% alkali treated), 9 (5% alkali treated), 4(9% alkali treated), 1(9% alkali treated) from Table 4.1 and one untreated composite we can find functional groups present in the composite. A broad peak is observed at 3327cm^{-1}-3300cm^{-1} for all the composites which is evidence of O-H group's stretching vibration for cellulose I. Stretching vibration of C-H for β cellulose is observed at 2914cm^{-1} to 2916cm^{-1}. CH_2 stretching vibration can be observed at 2836cm^{-1} to 2837cm^{-1} for hemicellulose [Vijay et al. 2020]. At 1725cm^{-1} C=O stretching vibration of lignin is only found in the untreated composite assuming it got partially or mostly removed from all the other alkali treated composite and did not show a noticeable peak. Bending peak at 1618cm^{-1} was observed due to water absorption of O-H group. From peak1589cm^{-1} to 1592cm^{-1} is observed for C=C-C aromatic ring stretch. CH_2 symmetric bending can be seen at peak 1478cm^{-1} only for untreated composite due to the presence of wax, hemicellulose and other impurities which are assumed to be partially or mostly removed in case of other alkali treated composites. From peak 1451cm^{-1} to 1460cm^{-1} corresponds to methylene C-H bending vibration. CH_2 symmetric bending vibration at 1412cm^{-1} is seen for untreated

composited because of Hemicellulose which is not noticeable for treated composites. Peak at 1375cm^{-1} corresponds to C-H bending in plane. Peak at 1164cm^{-1} corresponds to C-O-C symmetrical stretch vibration because of cellulose and hemicellulose. Peak at 1256cm^{-1} corresponds to C-O stretching vibration for acetyl group of lignin. Observed peak at 1034cm^{-1} for untreated composite corresponds to bending vibration of C-H and C-O groups of aromatic rings of cellulose. Peak at 997cm^{-1} corresponds to CH_2 bending vibration of cyclohexane ring. Peak observed below 650cm^{-1} corresponds to alkyne C-H bending vibration of lignin [Chun et al. 2013, Nandiyanto et al. 2019].

6.4 Thermal Properties

Thermal analysis is a test used to assess how a material would change chemically, physically, and structurally as a result of a temperature change. In general, the majority of chemical reactions, physical qualities, and structural changes are influenced by temperature, which is a basic state variable. Thermal analysis is a broad term that refers to any scientific or technical description of a material that uses temperature variation as an experimental parameter. However, the use of this word has historically been restricted to certain methods including thermogravimetric and calorimetric effects. The basic methods used in a thermal study are now generally acknowledged to be thermogravimetry (TG), its derivative (DTG), and differential scanning calorimetry (DSC) for determining heat flow.

From table 4.2, TG-DTG curve of specimen 3(5% fiber load, 75% cotton-25% areca volume fraction 5% alkali treated) and from figure 4.11, we can see the change of mass over temperature in TG as well as it's derivative in DTG curve. Minor mass change of 0.38% shows the moisture vaporization. 5% mass change occurred at 372^oC. At 498^oC amount of residual mass was 0.98%. So, it can be said that most of the composite was decomposed at that temperature. Specimen 5(15% fiber load, 50%cotton-50% areca volume fraction and 9% alkali treated) showed initial mass loss of 0.84% before 200^oC. Change of 5% mass occurred at 333^oC. Comparing with the previous specimen it is clearly evident that specimen 3 had more thermal stability up to this point. For specimen 5 another visible mass loss of about 14% was seen at 351^oC. This phenomenon occurred because of hemicellulose loss. Specimen 5 had 15% fiber load, therefore more hemicellulose and

moisture than specimen 3 with 5% fiber load. The DTG curve showed the highest decomposition temperature peak at 446^{o}C for specimen 3 and 450^{o}C for specimen 5 is the evidence of higher stability acquired at that temperature due to the availability of more fibers, which made a protective layer and inhibits process of thermal decomposition. The amount of residual mass at 498oC was 6.87% for specimen 5, which validates the claim. In the DSC curve series of exothermic and endothermic reaction can be observed. The melting temperature showed increased value for specimen 3 at 170^{o}C than for specimen 5 at 165^{o}C. So, melting temperature of polypropylene has been increased from 160-165^{o}C (melting temperature of commercial pp) to 170^{o}C due to fiber reinforcement.

Chapter-7

Conclusions and Recommendations

7.1 Conclusion

In the current study, a novel cotton-areca fiber reinforced polypropylene hybrid composite was created, experimentally characterized, and an RSM-designed mathematical model was developed to optimize the input parameters for a balanced composite combination. Based on the experimental study's findings and the work's statistical analysis, the following conclusion may be drawn.

i) Alkali treated novel cotton-areca reinforced polypropylene composite was fabricated by varying fiber loading, volume fraction of cotton-areca fibers and percentage of alkali concentration to characterize different characteristic of the fabricated composite

ii) Maximum tensile strength, flexural strength, tensile modulus, impact strength and hardness value were found for composite with 5% fiber load, 75% cotton-25% areca fiber and 5% alkali treatment.

iii) Maximum flexural modulus and minimum moisture absorption value were found for composite with 10% fiber load, 75% cotton-25% areca fiber and 9% alkali treatment

iv) Higher impact strength than most other natural fiber reinforced composite makes it a good option for automotive, construction, sports industries.

v) Scanning electron microscopic (SEM) images depicts improvement of fiber surface due to alkali treatment by removing no-load bearing components.

vi) Crystallinity Index improvement was observed due to hybridization of cotton-areca fibers with polypropylene compared to individual fiber or matrix by X-ray diffraction (XRD). Increase of crystal size from untreated

composite due to alkali treatment improves the hydrophobic nature of composite.

vii) Different functional groups were characterized from Fourier transform infrared spectroscopy (FTIR)

viii) Thermogravimetric analysis determined the thermal stability and different stages of decomposition peak temperature of the composite.

ix) RSM model was formulated using experimental data for tensile strength, tensile modulus, flexural strength, flexural modulus, impact strength, hardness and moisture absorption. From ANOVA table significance of the model, input parameters, interacting terms and square terms were determined. In most of the cases all three input variables were found to be significant whereas fiber loading being the most significant.

x) RSM generated prediction model was found to have higher accuracy with very low mean absolute percent of error.

xi) Optimization was performed using desirability function with a desirability value of 0.927.

7.2 Recommendations

i) Different polymer matrices (epoxy, polyester, polyvinyl, high-density polyethylene, and low-density polyethylene) can be utilized with various natural fibers (e.g., jute, sisal, banana, sugarcane, rice husk, and pineapple).

ii) The composites may be produced using a variety of fiber orientations, chemical treatments, fiber lengths, layer configurations, and manufacturing techniques.

iii) A variety of mechanical characteristics, including shear strength, fatigue resistance, and percentage of elongation, may be identified.

iv) The input parameters may be optimized using a variety of optimization algorithms, including Taguchi method, genetic algorithm, and particle swarm optimization.

v) Different machining operation like milling, drilling, grinding can be done to find out different surface roughness values for different composites.

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