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The modification of flax fiber and polypropylene composites by acetylation and its consequences on their physical qualities

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Abstract. Acetylation was used to modify flax fiber. Besides preparing modified flax fiber reinforced polypropylene composites, researchers looked at how acetylation affected the structure and characteristics of flax fiber. The acetylation reaction speed was increased with the help of the catalyst. Flax fiber was altered, and the results were characterized. Flax fibers were examined for a number of characteristics, including their surface shape, moisture absorption property, component content, degree of polymerisation, crystallinity of cellulose, and heat stability. Incorporating acetylation into the flax fiber production process significantly enhanced the fibre's surface shape and moisture resistance. Polypropylene composites reinforced with flax fiber (both modified and unmodified) were made. The fiber loading was 30 wt%. The composites' mechanical characteristics were studied. Researchers showed that the tensile and flexural strengths of composites increased up to a degree of acetylation of 18%, but afterwards dropped. It was discovered that the Charpy impact strengths of composites dropped as the acetylation level rose. It was discovered that the tensile and flexural strength parameters increased by anywhere from 20 to 35% when the coupling agent (maleated polypropylene - MAH) was added.

Keywords: *Reinforcements, acetylation, fiber characterization, mechanical qualities, and flax fiber.*

1. Introduction

To produce natural fiber-reinforced composites, researchers need a deeper familiarity with the chemical composition and surface adhesive bonding of natural fiber. Natural fibers are made up of a wide variety of components, including cellulose, hemicellulose, lignin, pectin, fat, waxes, and water-soluble compounds [1-3]. Even for the same kind of fiber, the composition might change depending on the growth conditions and testing procedures [4]. Incorporating natural fibers like cellulose, which is a semicrystalline polysaccharide with a high hydroxyl group content and hence a hydrophilic character, into hydrophobic matrices leads to a very weak interface and poor resistance to moisture absorption [5]. Poly(-hydroxyethyl) hemicellulose

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likely hydrogen-bonded to cellulose fibrils. Hemicellulosic polymers are branching, completely amorphous and have a substantially lower molecular weight than cellulose. Because of its open structure containing multiple hydroxyl and acetyl groups, hemicellulose is partially soluble in water and hygroscopic [6]. Lignins are the least water-absorbing component of natural fibers [6], while being amorphous, extremely complex, predominantly aromatic polymers of phenylpropane [7] units.

There is a strong attraction or contact between the hydroxyl groups of the fiber's components and water molecules, giving the natural fiber a high hydrophilicity. Initially occurring in the non-crystalline zone, fiber-water interactions have been seen to propagate to the crystalline region. location where crystals may be found. Hydration is a process in which hydroxyl groups that are easily accessible are essential to the absorption of water by hygroscopic substances like cellulose and hemicelluloss. The driving power for further absorption is provided by the exothermic reaction created when water molecules are absorbed by cellulose molecules to produce cellulose hydrate [8, 9]. Because of its extreme vulnerability to moisture, lignocellulosic fiber is seldom used as a reinforcing component in composites. Because of the hydrophilic character of natural fibers, their potential as reinforcing agents is generally dampened; chemical changes are thought to maximize the interface of fibers. In most cases, molecules with several functions are used as chemical coupling agents. Firstly, it reacts with hydroxyl groups in cellulose, and secondly, it

raise the acetylation level. Sulfuric acid, pyridine, potassium and sodium acetates, gamma rays, etc. are only some of the many catalysts that have been utilized in the past. Nevertheless, there are significant challenges associated with catalyst use. Damage to the fibre structure is known to come from hydrolysis of cellulose when strong bonding [18].

It has been claimed that acetylating jute fibers may reduce their moisture absorption by roughly 50%, and that acetylating pine fibers can reduce it

reacts with functional groups in the matrix [10].

Chemical alterations of fibers, including acetylation, methylation, cyanoethylation, benzoylation, permanganate treatment, acrylation, etc., may decrease the amount of moisture absorbed by the fabric [11–15].

The esterification process known as acetylation, which results in the plasticization of cellulosic fibers, is widely used in the textile industry. The replacement of hydroxyl groups in the cell wall polymer with acetyl groups modifies the characteristics of these polymers, making them hydrophobic, which may strengthen the cell wall against moisture and enhance dimensional stability and resistance to environmental deterioration [16].

Moreover, acetylation is one of the most investigated reactions of lignocellulosic materials. Partially acetylated cellulose products, such as acetate rayon fibers and cellulose acetate plastics, entered the market in the early 1900s after their discovery in 1865. Acetylating wood flour and sawdust was first attempted in 1928. Methodology relies on reacting acetyl groups (-CH_3) with hydroxyl groups (-OH) found in the fibre components (CH_3CO–). In a homogeneous phase (when cellulose is dissolved), the reaction is known to proceed to complete esterification of all three hydroxyls of anhydro-D-glucose, but in the case of fibers and wood, the reaction is heterogeneous. The hydroxyl groups of the fiber are assumed to be esterified as a result. As a result, the end result may be quite variable. Catalyst speeding is essential in such circumstances.

mineral acids or acid salts are used [17]. Therefore, acetylation of lignocellulosic fibre requires careful catalyst selection and optimization. Hydroxyl groups in lignin, hemicelluloses, and amorphous cellulose are the reactive ones in the fiber. This is due to the inaccessibility of the hydroxyl groups un areas of the crystal with tight packing and strong interlock by up to 65% [10].

Researchers Seena et al. [12] observed that acetylation improved the tensile strength, tensile modulus, and impact strength of banana fiber reinforced phenol formaldehyde composites compared to untreated banana fiber composites.

Acetylation's impact on natural fiber composites (cotton, rayon, wood with poly- styrene as matrix) was researched by Liu et al., who demonstrated, using the micro-debonding test, that acetylated fibers had higher interfacial shear strength. In addition, they found that acetylation increased the fibers' surface free energy [19].

Researchers Zafeiropoulos et al. [17, 20] found that aectylation of flax, hemp, and wood fibre removed non-crystalline elements of the fibers, modified surface topographic features, adjusted fibre surface free energy, and enhanced stress transmission efficiency at the interface.

In this study, we examined the impact of acetylation on flax fiber properties, including composition, surface, crystallinity, polymerization, moisture absorption, and thermal stability. We also looked at how acetylation affected the properties of flax fiber reinforced polypropylene composites. Experimental

1.1. Materials

Germany's Mühlmeier GmbH supplied the green flax fiber (6-8 mm) used in this study.

We obtained the following chemicals from Merck KgaA, Darmstadt, Germany: acetic anhydride, toluene, perchloric acid, sodium hydroxide, hydrochloric acid, ethanol, sulphuric acid, acetic acid, sodium chlorite, acetone, iodine, potassium iodine, sodium sulphate, sodium thiosulfate. The quality of the chemicals used in the analysis was quite high.

As a coupling agent, we employed a maleic anhydride-polypropylene copolymer (Licomont AR 504 FG) that is commercially available and has an acid number of 37-43 mg KOH/g. It was sourced from the Frankfurt, Germany headquarters of Clariant Corporation. The melting point of this material was 153 degrees Celsius, and its density ranged from 0.89 to 0.93 grams per cubic centimeter. A weighted proportion of 5% fibre was contributed by this ingredient.

1.2. Methods

1.2.1. Acetylation process

After an hour of soaking in demineralized water, the flax fibers were filtered and added to an acetylating solution in a round-bottom flask. You'll need 250 ml of toluene, 125 ml of acetic anhydride, and a pinch of catalyst perchloric acid (60%) to make an acetylating solution. Acetylation took place between one and three hours at a temperature of 60 degrees Celsius. Following the process of modification, the fiber was rinsed in distilled water on a regular basis until it was no longer acidic. Following an appropriate period of air drying, the modified flax fibers were tested.

Analyzing Synthetic Fibres with a 2.2.2-Point Modification Acetylation Saturation:

Saponification techniques were used to quantify the presence of acetyl groups. The 0.5N sodium hydroxide solution was used to saponify the modified flax fiber. After saponification, 0.5N hydrochloric acid was used to titrate the surplus alkali. The degree of acetylation may be determined using a similar procedure applied to untreated flax fibre.

materials that can be extracted

The 5 g of air-dried material was measured into an extraction thimble and transferred to the Soxhlet apparatus. The extraction procedure was place over the course of five hours, using a solvent consisting of equal parts ethanol and toluene. The material was dried at 60 degrees Celsius to constant weight after being washed with ethanol and hot water after extraction. Each sample was subjected to the procedure used to determine the extractibles as a percentage of the oven-dried test sample.

Lignin concentration

A total of 15 milliliters of 72% sulphuric acid was applied to a flask containing 2 grams of extracted material. After the mixture had been sitting at 25°C and being agitated for 2.5 hours, 200 cc of distilled water was added and the temperature was kept at that. Next, the ingredients were brought to a boil and simmered for two hours before being chilled. The lignin was transferred to the crucible after 24 hours and rinsed with hot water until it no longer contained any acid. Lignin was collected, then dried at 105 degrees Celsius, weighed after cooling in a desiccator. Multiple cycles of drying and weighing were performed until a stable weight was achieved.

Total amount of holocelluloses

Weighing out three grams of air-dried flax fiber, we put it to an Erlenmeyer flask along with 160 ml of distilled water, 0.5 ml of glacial acetic acid, and 1.5 g of sodium chloride in three separate increments. To a flask containing 0.5 ml of glacial acetic acid and 1.5 g of sodium chloride, we added 75°C and an hour in a water bath. Sodium chloride and acetic acid were added twice an hour. The flask was put into an ice bath and chilled to less than 10 degrees Celsius. Following filtration and washing in acetone, ethanol, and water, the holocellulose was finally dried in an oven heated to 105 degrees Celsius before being weighed.

α-cellulose content

A beaker containing 2 grams of holocellulose was treated with 10 milliliters of a sodium hydroxide solution (17.5%). In order to soak the fibers in sodium, a glass rod was used to agitate them.

aggressively with a hydroxide solution. Thereafter, the mixture was maintained at 20 °C while sodium hydroxide solution was added periodically (once every five minutes) for half an hour. An hour after adding 33 ml of distilled water to the beaker, we checked on it. The remaining holocellulose was filtered and placed in the crucible, where it was washed with a series of solutions, including 100 ml of sodium hydroxide (8.3%), 200 ml of distilled water, 15 ml of acetic acid (10%), and finally more water. The -cellulose-containing cru-targeted substance was dried and then weighed.

High Levels of Hemicellulose

Equation (1) was used to determine the hemicellulose concentration in flax fibre.

Holocellulose minus -celluloses equals hemicelluloses (1)

Calorie content of cellulose

We used capillary viscometry to determine the molecular weight of cellulose using the Cuoxam technique. It was discovered that cuoxam solution did not fully dissolve the cellulose. The solution was filtered and its viscosity was tested beforehand. This procedure was carried out at room temperature, and it is appropriate for substances with viscosities between 0.1 and 2.0. There has been some accounting for temperature and damage adjustment. Equation (2) was used to get the typical molecular weights (DP = polymerization degree):DP = $\eta/[1 + (0.28\cdot\alpha)]\cdot(1000/6.1C)$

(2)

where η is the specific viscosity, C is the concen- tration of cellulose materials in cuoxam solution.

Degree of crystallisation of cellulose

Cellulose crystallinity was measured using the iodine absorption technique. Two milliliters of an iodine solution (10 milligrams of iodine and eighty milligrams of potassium iodide in one hundred milliliters of demineralized water) and one hundred milliliters of a saturated sodium sulfate solution were added to thirty-three milligrams of treated flax fibers in a beaker. Once the beaker's temperature was set to 20 degrees Celsius and the sodium sulphate solution was added, it was placed in a cool, dark room for one hour. It was possible to calculate the amount of iodine still present in the solution.

using a 0.03 M sodium thiosulfate titration (starch as indicator).

Thermo gravimetric analysis (TGA)

TA Instrument's supplied thermal gravimetric analyser (TGA-Model TG 50) was used to compare treated and untreated flax fibers. We have collected a 20-milligram sample of each fiber type for testing. In a nitrogen atmosphere, the samples were heated at a constant rate of 20 K/min from 25 to 500 °C. For each sample, we ran the analysis twice to ensure accuracy.

Transmission electron microscopy

Using a scanning electron micro-scope (SEM), MV2300, CamSan Electron Optics, we looked at the morphology of treated and untreated flax fibers. Before the investigation, flax fibers were affixed to a metal surface using a specific glue and sputter coated with gold.

Method 2.2.2: Mixer-Injection Molding for Composites

High-speed cascade mixing was used to combine treated or untreated flax fibers with polypropylene (Henschel heat-cooling mixer system, type HM40-KM120). Before being combined, flax fibers were dried at 80 degrees Celsius for 24 hours (moisture content 1%) in an oven with circulating air. Once the flax fiber and polypropylene (in a ratio of 30% each) reached the melting point of polypropylene (173°C) in a hot mixer, the agglomeration granules were moved to a cold mixer, where the cold water brought them down to room temperature. After a second round of drying (80 degrees Celsius for 24 hours), the cold agglomerate granules were ready for the injection molding stage of sample preparation. The dry agglomeration was injected into a mold at 80 degrees Celsius, and the injection pressure was set at 20 kilograms per square millimeter.

1.2.2. Characterization of composites

In order to learn more about composites, we put them through a battery of tests including tensile, flexural, and impact. Flexural and tensile tests were conducted at a test speed of a Zwick UPM 1446 machine at a rate of 2 mm/min in accordance with EN ISO 527 and EN ISO 178. All experiments were conducted at a constant relative humidity of 50% and a room temperature of 23 degrees Celsius. Ten notched samples were tested using a Charpy impact machine by Zwick in accordance with EN ISO 179. The Charpy impact strength was determined for each situation using a standard variation of 5% (drop weight).

The Third and Final Part: The Results and Discussion

Acetylation, Step 3.1

What Happens When a Catalyst Is Used? 3.1.1

The catalyst % used to modify the flax fibers was varied, and the process was carried out at a temperature of 60 degrees Celsius. Using a catalyst and not using a catalyst for the acetylation process was analyzed. As shown in Figure 1 [17], the acetylation reaction rate increased in the presence of catalyst, whereas the reaction kinetic of the acetylation process was slower and depended on the moisture level of the fiber. The reaction rate was observed to rise by almost thrice after one hour when 0.026% catalyst was added to the acetylation procedure. In the presence of 0.052% catalyst, the acetylation level of flax fiber may increase up to 40-45% in about three hours. When the catalyst concentration is higher than 0.052%, the kinetic of the reaction becomes extremely rapid and abruptly destroys the fiber, making it impossible to regulate the acetylation process.

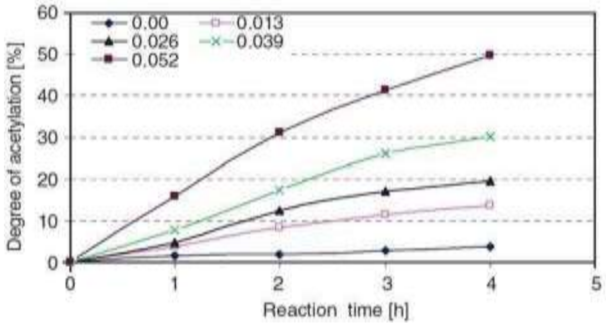


Figure 1. Influence of catalyst concentration on degree ofacetylation

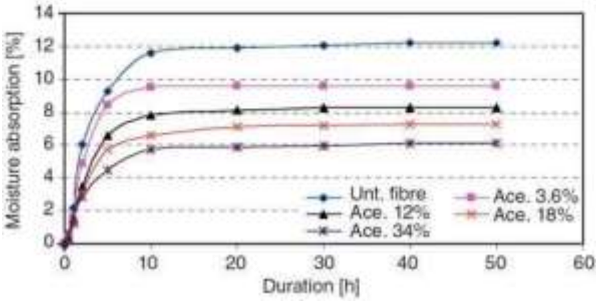


Figure 2. Influence of acetylation of flax fibre on moisture absorption property at 65% relative humidity

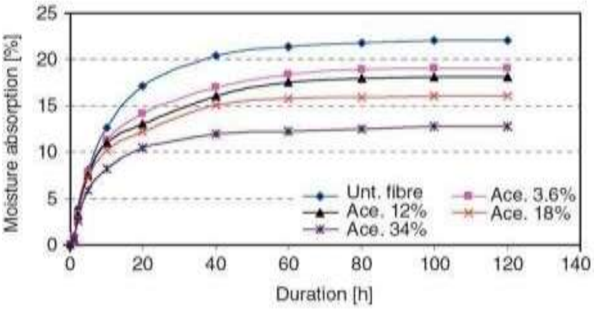


Figure 3. Influence of acetylation of flax fibre on moisture absorption property at 95% relative humidity

Analysis of Fibre Properties

Adsorption of Water (2.3.1)

Illustrations of the effect of flax fiber acetylation level on moisture-absorbing capacity are shown. seen in Figures 2 and 3 at 65% and 95% relative humidity, respectively. When compared to untreated flax fiber, the moisture absorption rates of flax fiber with an acetylation level of 3.6%, 12.6%, 18%, and 34% were about 21.3%, 32%, 40%, and 50% lower, respectively, at a relative humidity of 65%. The moisture absorption characteristics of acetylated flax fiber decreased by 14, 18, and 42% from those of untreated flax fiber when exposed to 95% RH. As the acetyl concentration of the fiber increased, it was found that its hydrophilicity reduced, and hence its ability to absorb moisture.

1.2.3. Fibre components

Figure 4 depicts how acetylation changes the make-up of the fibers. Slowly decreasing cellulose contents were found at 12% degree acetylation owing to degradation of cellulose, increasing amorphous content, and acetylated hemicellulose deposition on the fiber surface [20]. This was caused by the extraction of lignin and extractibles from the fiber. Because of the lignin's solubility and the extractives' concentration, it was also shown that the content of lignin continuously dropped up to 34% acetylation.

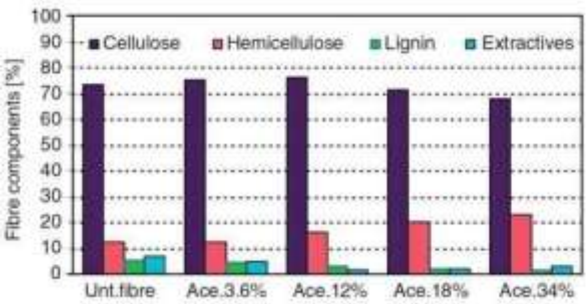


Figure 4. Influence of acetylation of flax fibre on the con-tent of fibre components

decreased till 12% acetylation and then increased slowly which is because of cellulose degradation. On the other hand hemicellulose contents were found to increase with increasing degree of acetyla-tion.

1.2.4. Degree of polymerisation and crystallinityof cellulose

The effects of the acetylation on the degree of poly- merisation and crystallinity of cellulose illustrated in Figure 5. It was observed that the degree of poly-merisation slowly decreased with increasing degree of acetylation till 18%. It is because of increase amorphous (low molecular weight acetylhemicellu- lose) content. After 18% acetyl content the degree of polymerisation decreased sharply which because of vigorous degradation of cellulose.

From the Figure 5 it is also observed that the degree of crystallinity a little bit increased initially with respect to degree of acetylation which is because ofremoval of lignin and extractibles. After that the

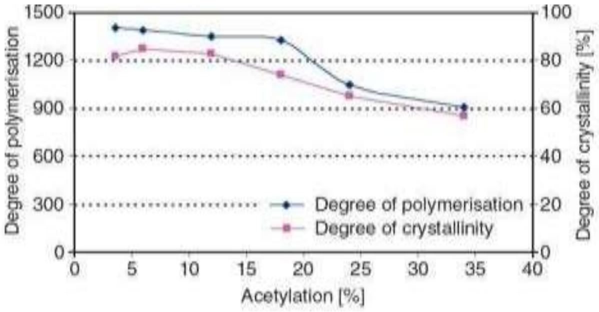


Figure 5. Influence of acetylation of flax fibre on the degree of polymerisation and degree of crys-tallinity of cellulose

Deposition of acetylated amorphous components on the surface of cellulose led to a reduction in the crystallinity of the cellulose, as measured by the degree of acetylation [20].

Consistency in Temperature 2.2.5

Deposition of acetylated amorphous components on the surface of cellulose led to a reduction in the crystallinity of the cellulose, as measured by the degree of acetylation [20].

1.2.5. Morphology

Figure 6 depicts the surface morphology of treated and untreated fibers. Figure 6a, 6b shows that an unprocessed fiber's surface is rough, with waxy and projecting features. Viewed in Figure 6c and 6d is the surface morphology of treated fiber. The interaction with acetyl removes the wax and cuti- cle in the surface, resulting in a smoother surface, on the acetylation treatments. Fibrillation is discovered to happen when binding materials are eliminated and micropores form in the treated fibers. Figures 6e, 6f show that when acetylation levels rose, so did fibrillation, but fibers were damaged and cracked.

1.3. Mechanical properties of composites

1.3.1. Tensile properties

Figure 7 shows the degree of acetylation of flax fibre on tensile properties of composites. Tensile

Table 1. Thermal stable temperature of treated and untreated flax fibre

	Untreated	Degree of acetylation [%]				
		3.6	**6**	**12**	**18**	**34**
Stable temperature [°C]	319	326	329	334	341	360

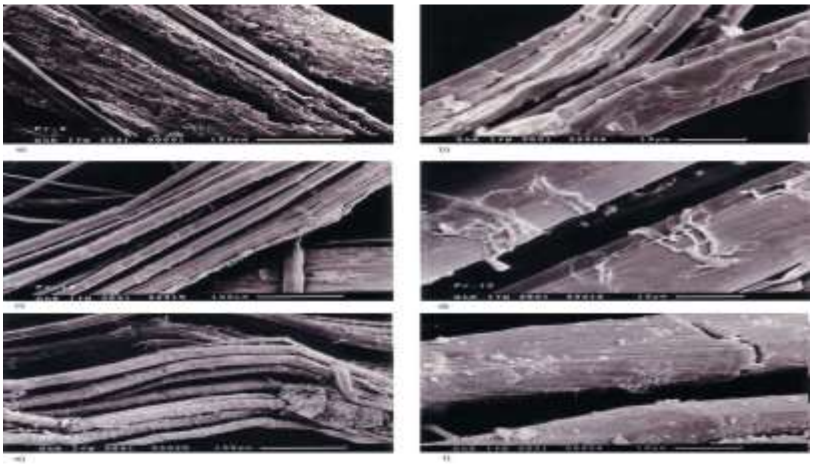


Figure 6. SEM photograph of flax fibre; (6a, 6b) untreated flax fibre surface, (6c, 6d) acetylated flax fibre at degree of acetylation 18% and (6e, 6f) fibre damage at high degree of acetylation

We discovered that the strength of composites increased with acetylation level up to 18%, but subsequently declined with further acetylation level. The conversion of some of the hemicellulose to acetylated hemicellulose, together with the minor increase in cellulose content and the elimination of the lignin and extractibles, may account for the observed improvement in tensile strength.

lose. The tensile strength of composites was shown to rapidly decline when flax fiber was acetylated at 18%. The fibers may have developed interior breaks as a result of cellu-loss degrading. Acetylation of flax fiber was shown to reduce wax on the fiber's surface, improve the fibre-matrix interface, and lengthen the lifespan of the fibre.

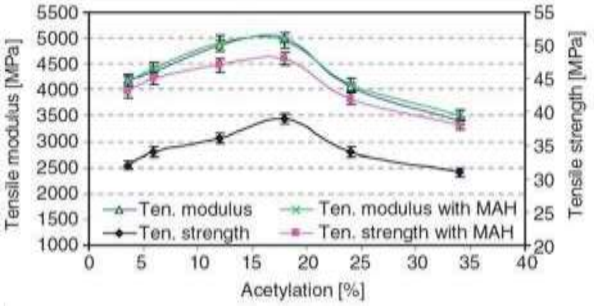
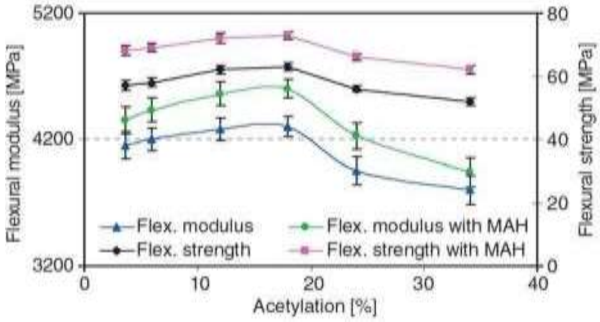


Figure 7. Influence of acetylation of flax fibre on tensileproperties of composites

enhancements in surface free energy that are conducive to enhanced composite characteristics. Similar tendencies were seen when MAH was added, and the tensile strength increased by 20%-35% depending of the acetylation level. This occurs because ester bonds are formed between the fiber and the polypropylene.

1.3.2. Flexural properties



1.3.3. For a material to be considered flexural strong, it must be able to resist forces that are perpendicular to its longitudinal axis and bend it. Compressive and tensile stresses act together to cause deformation under a flexural load. The specimen is deflected until the outer fibers of a polymeric material break under a flexural force. Figure 8 depicts how acetylation modifies the flexural properties of flax. We find that acetylation can be used to intentionally boost flexural strengths up to around 18%, after which they decline precipitously. The flexural modulus was shown to rise somewhat up to 18% acetylation, before gradually decreasing with further acetylation. Removal of the outer surface is probably responsible for the enhanced flexural characteristics of treated fiber composites;

Figure 8. Influence of acetylation of flax fibre on flexuralproperties of composites

maximize the physical and chemical changes generated by fiber treatment, including an increase in cellulose content and effective surface area, as well as interfacial adhesion. Acetylation also causes flax fiber to fibrillate and decrease in diameter, both of which may affect the composites' modulus qualities. Cracking and damage to the fibers were seen as the acetylation level increased, suggesting that above a certain acetylation level, the characteristics of the composites might be compromised. Due of enhanced interfacial contact, MAH boosts flexural strengths by as much as 20%. However, when considering standard deviation, the effect of MAH on flexural modulus is less noticeable.

Impact characteristics as measured by the Charpy impact criterion

Many elements affect the impact strength of a composite, such as the reinforcement's toughness qualities, the interfacial region's composition, and the amount of frictional effort required to separate the fiber from the matrix. Determining the composite's toughness relies heavily on the interface region's composition.

Figure 9 displays the relationship between the acetylation degree and the notched Charpy impact strength of flax fiber- PP composites. The amount of energy absorbed by a material during fracture may be measured using the Charpy impact test, which is a standardized high strain-rate test. The amount of energy that is absorbed during a collision is used as a tool for studying the brittle-ductile transition [21].

Debonding occurs as a result of fracture propagation at the fibre-matrix contact, as shown by Ray et al. [22]. Because of this, the composites' ability to absorb energy is much improved.

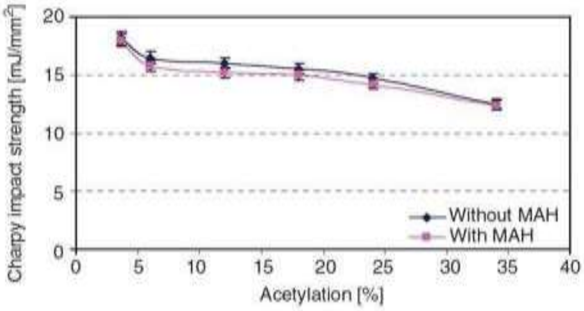


Figure 9. Influence of acetylation of flax fibre on charpyimpact strength of composites

greater resistance to impact fatigue is achieved because to the huge new surfaces created and frictional work resulting from differential displacement between matrix and fibre.

It can be seen from the figure that the notched Charpy impact strength drops gradually with increasing acetylation degree, which is related to the strong interface and the reduction in toughness of the composite [12]. There was no observable change caused by the addition of MAH-PP, and in fact, it was discovered that the impact characteristics were somewhat diminished. Increased brittleness in the matrix material and local internal deformation in the composite material might account for this.

2. Conclusions

The purpose of this research was to examine how acetylating flax fiber modified the fibre's qualities and how those modifications affected the properties of the composites they were used to make.

Conclusions: - Even a very modest concentration of catalyst may significantly alter the pace and extent to which acetylation occurs.

The acetylation of flax fibers may reduce their moisture-absorption capacity by as much as half.

Due to acetylation, flax fibers have a different shape and different components.

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In a given temperature range, acetylation improved thermal stability.

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Strength qualities were shown to be improved by around 25% when comparing acetylated flax fiber composites to untreated fiber composites, with the highest tensile and flexural strengths being seen at 18% degree.

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The tensile strength of composites increased by 20-35% when MAH was added, depending on the degree of acetylation.

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