RESEARCH ARTICLE

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Surface Modifications of Natural Fibers for Use in Preparation of Biocomposites

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Abstract

This century has observed unusual improvements in green materials through the development of biocomposites due to anxieties about the atmosphere and sustainability issues. These biocomposites can be effectively disposed of at the final existence without harming the environment, which is impossible with man-made fiber-based polymer composites. The natural fiber is not a problem-free option, and they maintain some shortfall features that are the considerable moisture assimilation and very anisotropicity. This review paper intends to give a short outline of various surface modification methods to improve the fiber–matrix adhesion influencing the significant enhancement of the mechanical properties of the biocomposites.

Keywords: Surface treatment, Bio-composites, Interfacial bonding, Natural fibers, Properties, Fiber/matrix adhesions

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Introduction

In recent years, for making low-cost engineering materials, using the reinforcement natural plant fibers in polymer composites has gained much interest. The rising concern towards environmental issues, new environmental regulation, and consumer pressure have forced manufacturing industries, mainly automotive, construction, and packaging, to search for new polymer composite materials filled with natural-organic fillers, which are coming from renewable sources that can substitute for conventional non-renewable reinforcing materials such as glass fiber.[1-7] Availability in large amounts, lightweight, and free from health hazards, biodegradable, specific strengths, modulus, economic viability, low density, reduced tool wear, and enhanced energy recovery are the acceptable advantages of natural plant fibers over traditional glass fibers.[8-10] The commonly available natural fibers are flax, jute kenaf, hemp (extracted from bast), sisal pineapple, palf (extracted from leaf), cotton, kapok (extracted from seed), coir (extracted from fruit), bamboo, elephant grass (extracted from stalk), etc. However, natural plant fiber-reinforced polymeric composites also suffer from some limitations such as poor bonding between the natural hydrophilic fibers and hydrophobic thermoplastic and thermoset matrices, poor moisture resistance, especially absorption, and low strength compared to synthetic fibers materials. The propensity of water absorption of natural fiber polymer composites is a severe trouble, especially for their potential outside applications.

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Natural fibers are not an issue-free alternative and have specific deficits in properties. Their primary beneficiary structures (cellulose, hemicelluloses, lignin, gelatin, and waxy substances) permit moisture ingestion from the environment, prompting helpless holding with the matrix materials.[11] The chemical structures of the matrix and fibers vary, and couplings between these two phases are challenging. This causes ineffective stress transfer throughout the interface of the composites and although the proper chemical and physical treatments can improve the adhesion between the matrix and the fiber composite.[12-15] Consequently, certain chemical treatments on the surface of natural fibers are unquestionably required. These treatments are generally founded on reagent functional groups equipped to respond with the fiber structures and change their composition proportion.

Subsequently, different chemical treatments were carried out to increase the moisture resistance by way of improving their adhesion and reinforcing natural fibers with a polymer matrix. These treatments are alkaline treatment,[16] acetylation,[17] benzoylation treatment,[18] silane treatment,[19] acrylation,[20] acrylonitrile grafting,[21] peroxide treatment,[22] permanganate treatment,[23] isocyanate treatment,[24] etherification of natural fibers,[25] graft copolymerization of natural fiber,[26] sodium chlorite treatment[27] of natural fibers, sodium bicarbonate treatment,[28] acetic anhydride modification with maleic anhydride,[29] and styrene titanate treatment.[30]

Chemical Treatment

Alkali Treatment

Alkali treatment (mercerization) commonly uses chemical treatment to modify fiber structure by treating sodium hydroxide (NaOH). Natural fiber absorbs moisture in the amorphous region of hemicellulose, lignin, and cellulose constituents due to hydroxyl groups.

Alkali treatment includes eliminating the hydroxyl group of fiber in the reaction with NaOH to produce water molecules (H-OH). Thereby, Na-O- combines with the cell wall of fiber to generate the fiber-cell-O-Na groups, as provided in equation 1.[31] Alkaline treatments of fiber diminish the moisture contained hydroxyl groups and thereby the weakened hydrophilic nature of the fibers. The chemical reaction of the fiber–cell, and NaOH is represented in Scheme 1.

The NaOH concentration, treatment times, and temperature of treatment play a vibrant role in achieving the fiber's optimal effectiveness. However, a high soluble base concentration may cause an abundance end of covering materials from the cellulose surface and delignify the fiber significantly, adversely affecting the strength of the fiber and weakening or damage to the fiber structure. Accordingly, alkaline handling straightforwardly impacts the cellulosic fibril, polymerization level, and the extraction of lignin and hemicellulose compounds.[43]

Scheme 1

Table 1: 3 Recent works on alkali-treated fiber composites

S. No	*Fiber matrix*	*Applied treatment*	*Results*	*Ref.*
1	Flax-Epoxy	NaOH treatment	30% increase in tensile strength and modulus	[32]
2	New cane- polyester	2–8% NaOH for 4 h	6% alkali treatment reported maximum tensile strength	[33]
3	Hemp- Euphorbia	0.16% NaOH for 48 h	30% tensile strength was increased and doubled the shear strengths.	[34]
4	Jute- Vinylester	5% NaOH for various time	4 hour alkali treated composite accounted for 20% and 19%increase in flexural strength and modulus	[35]
5	Coir-polyester	5% NaOH treatment for 72 h	Flexural and impact strength was increase by 40% with respect to untreated fiber	[36]
6	Hemp- polyester	8% NaOH treatment for 3h	27% increase in flexural strength & 26% increase in flexural modulus	[37]
7	Biofiber glass polyester	5, 10% NaOH treatment for 1h	5% alkali treatment obtained optimum tensile strength	[38]
8	Alfa fiber polypropylene	10% for 24 h	Improvement inYoung's modulus and tensile strength by about 23 and 16%, respectively	[39]
9	Sisal-epoxy	10% NaOH	Alkali treatment increases (i) fiber strength and (ii) The adhesion between the fiber bundles and the matrix.	[40]
10	Sisal-Polycaprolactone	10% NaOH For 24 and 48 h	Increasing elastic modulus obtained by increasing reaction time	[41]
11	Bagasse-polyester	1, 3, 5% NaOH 13%	Improvement in tensile strength, 14% in flexural strength and 30% in impact strength of composite was observed for 1% NaOH treatment.	[42]

Benzoylation Treatment

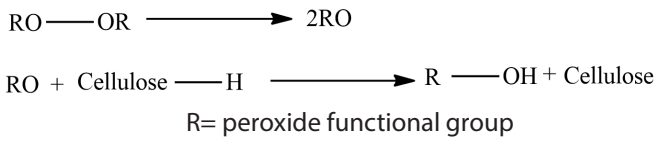
In benzoyl treatment, benzoyl chloride was utilized to deteriorate the hydrophilicity of fiber and strengthen the adhesion bonding at the collaboration between the fiber and matrix.[43] Hydroxyl groups are attached with cellulose by excluding constituents like waxes and lignin from the fiber surface. Next, OH groups of the fiber are displaced in the benzoyl group, and it joins to the cellulose. Results explained that hydrophobicity creates on the fiber surface and grows bond with the matrix. The chemical reaction is described in Scheme 2.

Scheme 2

According to Shanmugam and Thiruchitrambalam[44] Palmyra palm-leaf stalk fiber was pre-treated with NaOH and agitated benzoyl chloride for 15 minutes modulus, and the tensile strength of the composite increased by 60% with the treated fiber. This treatment was applied to low-density polyethylene-based, and its flax fiber composites were developed.[45] The finding found better moisture resistance and tensile strength properties due to superior interlocking between fibers and matrices. Wang[46] carried a similar process execution to boost the interfacial adhesion of flax fiber with polyethylene. Benzoylated sisal fiber composite has been postulated by Nair et al.[43] to investigate the consequence of benzoylation. It was evaluated that the thermal stability of treated sisal composites was superior compared to the untreated ones. Joseph et al.[47] used sodium hydroxide and benzoyl chlorite solution to treat sisal fibers and improved hydrophobicity after the treatment. Mathew and his co-authors[48] fabricated isora fibers natural rubber composites, and chemical treatments saw improved compatibility because of the reduction in the concentration of polar components on the surface. Dhanalakshmi et al.[49] performed different kinds of treatments such as benzoyl chloride treatment, potassium permanganate treatment, and acrylic acid treatment to enhance the performance of the composites by improving the adhesion of areca fiber with a natural rubber matrix. Kumar and Rajesh[50] analyzed the influence of alkali, permanganate, benzoylation treatment on banana fiber to prepare the composite using natural rubber as a matrix. The wear resistance, compressive strength, and hardness of the composites increased with better interphase properties after treatment. Jothibasu et al.[51] studied a better performance of flax/epoxy composites after HCl, alkali, and benzoyl treatment.

Peroxide Treatment

In this treatment, fiber and matrix interface features were improved by reacting the peroxide-free radical group with the hydroxyl group of natural fibers. As a result, the peroxide-induced grafting of polyethylene holds to the fiber surface. Besides, the moisture absorption capability of the diminished fiber and thermal stability improves. The reaction is specified in Scheme 3.

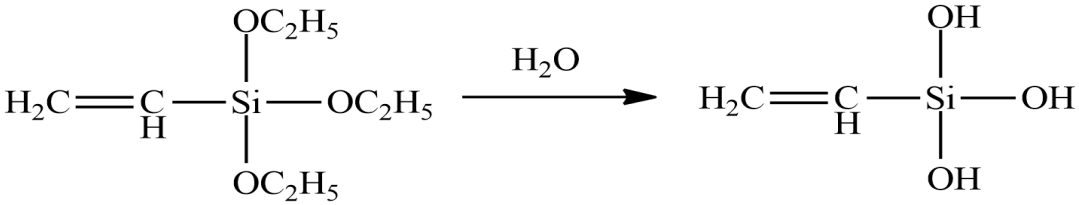


Scheme 3

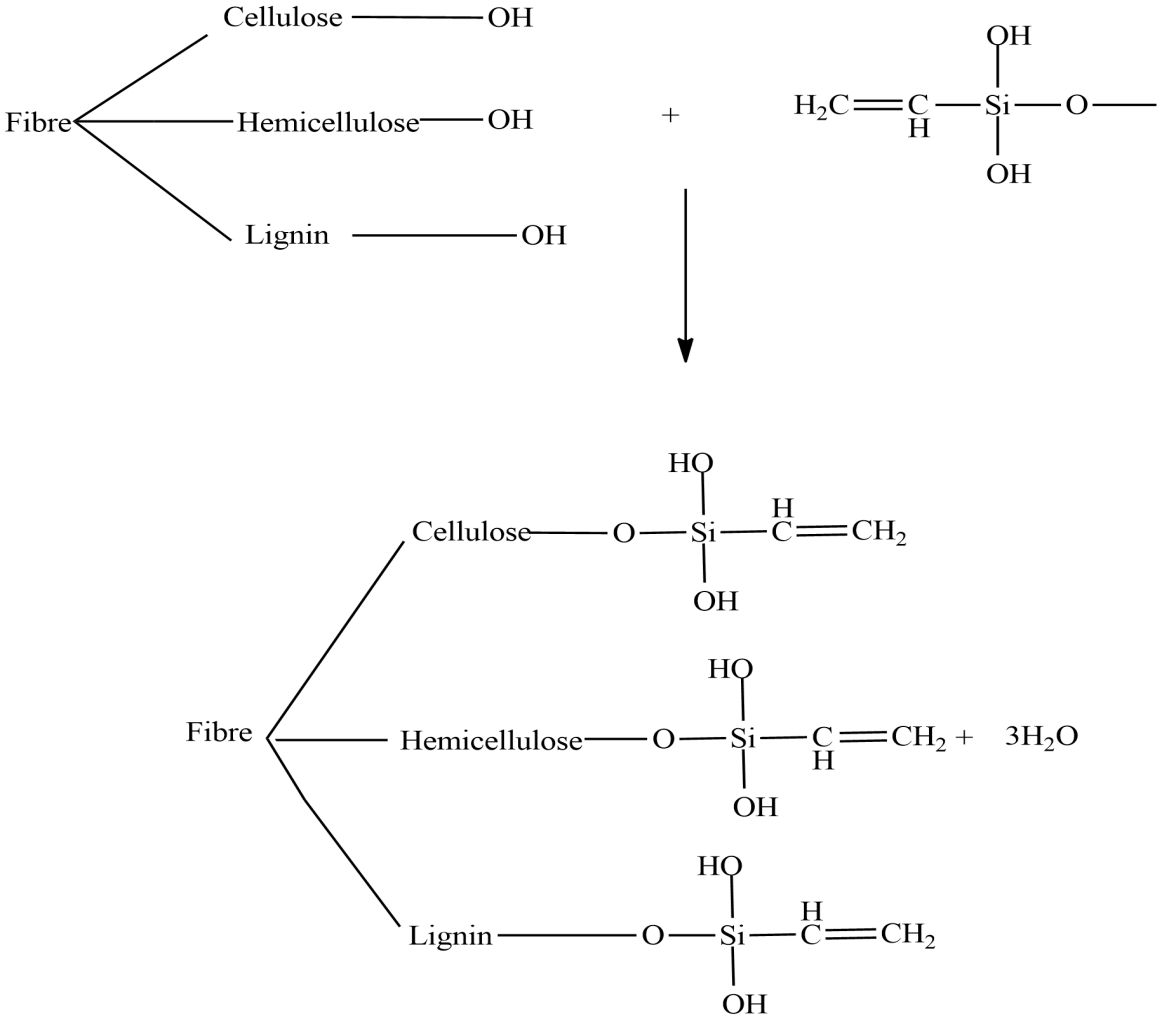
Joseph et al.[47] used investigated sisal fibers with varying concentrations of benzoyl peroxide and dicumyl peroxide (DCP). It was evaluated that after treatment, the tensile strength of polyethylene-based sisal composites enhanced up to an optimal 6% concentration of benzoyl peroxide and 4% DCP treatments after that, tensile strength values remain unchanged. Sood et al.[52] studied the outcome of peroxide treatments on tensile and flexural properties of sisal fibers/ high-density polyethylene. George et al.[53] reinforced pineapple-leaf fiber into low-density polyethylene to examine the effect of peroxide treatment. Treatments such as alkali, acetylation, permanganate, and peroxide were conducted on carnauba fibers by Melo et al.[54] Fiber–matrix adhesion was improved after peroxide treatment, as confirmed by SEM observations. Asaithambi et al.[55] prepared the hybrid composites using banana and sisal fibers with polylactic acid subjected to benzoyl peroxide treatment. Benzoyl peroxide treated fibers enhanced the compatibility between the fibers and PLA matrix through cross-linking. Manaila et al.[56] analyzed the effects of benzoyl peroxide on hemp fibers as well as fabricated natural rubber-based composites. Tayfun et al.[57] decreased the water uptake capacity of flax fiber polyurethane composite by this treatment. Lopattananon et al.[58] investigated the performance of pineapple leaf fiber–natural rubber composites after alkali treatment at varying concentrations (1, 3, 5, and 7%) and benzoyl peroxide treatment at varying concentrations (1, 3, and 5%).

Silane Treatment

Silane is utilized as a coupling agent to modify the surface of the fiber. Silane molecules form a chemical link between the matrix and the fiber through the siloxane bridge. For the period of the treatment process of the fiber, three stages of hydrolysis, condensation, and bond formation take place. In the hydrolysis process, silanol is formed by silane in fiber moisture given in the equation.[59] For the duration of the condensation process, one end of silanol reacts with the matrix functional group, and the different end reacts with the cellulose hydroxyl group. This process provides the hydrocarbon chain that restrains the fiber swelling into the matrix by which molecular continuity occurs across the composite interface, and fiber-matrix adhesion improves. Silane reaction on fiber is given in Scheme 4.



Scheme 4a



Scheme 4b

Seki et al. [60] evaluated the comparative study on the consequence of alkali and silane treatments against the flexural properties of treated jute-epoxy and treated jute-polyester composites. It was noted that silane treatment provided superior strength and modulus properties compared to the alkali treatment in both jute polyester composites and jute-epoxy composites. Asim et al.[61] modified the pineapple leave fibers and kenaf fibers by 6% NaOH and 2% silane performed fibers to enhance interlocking with polymer matrices. Results reveal that tensile modulus and tensile strength of silane treated pineapple leaf fiber composite increased owing to the exclusion of lignin and hemicelluloses and the effectiveness of silane treatment. Panyasart et al.[62] performed a similar kind of work wherein alkaline and silane treatments were conducted on pineapple leaf fibers. The study mentioned that silane-treated composites show a hike in thermal stability and mechanical properties compared to alkali-treated ones because mechanical interlocking by silane treatment was more efficient for improving fiber-matrix interfacial adhesion. Sreekala et al.[63] modified oil palm fibers by this treatment to determine their water uptake behavior, and the results showed that after treatment, the hydrophilicity of fibers and the water uptake was reduced owing to variation in physical and chemical changes on fibers surface. Alix et al.[64] applied silane and styrene treatments on flax fibers, and moisture resistance of its polyester resin

composites was found to increase. Pothan et al.,[65] studied the influence of silane treatment on the dynamic behavior of banana fiber polyester composite mechanical properties. Silane treatment enhanced the storage modulus but lower damping behavior. Yu et al.[66] studied alkali and silane treatment on dynamic mechanical properties of ramie fiber reinforced polylactic acid composites. Storage modulus increased after treatment by cause of boost in stiffness which allows more stress transfer from resin to fibers.

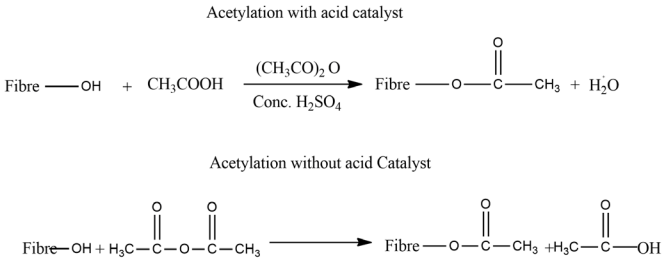
Permanganate Treatment

Permanganate treatment of cellulosic fibers is carried by potassium permanganate ($KMnO_4$) in an acetone solution. This treatment forms highly reactive permanganate (Mn^{3+}) ions to reacts with the cellulose hydroxyl groups and forms cellulose–manganate to boost interlocking; thereby, better adhesion is formed.[67] The reaction between fiber–OH group and potassium permanganate is specified in Scheme 5. Sreekumar et al.[68] fabricated composite using treated sisal fiber as reinforcement and polyester as a matrix. Permanganate treatment was done on alkali-treated sisal fiber, and it was concluded that permanganate treated sisal fiber reinforced polyester composite has a superior value of flexural strength than untreated sisal fiber composites. The reason for this was better interaction between fiber and resin. Paul et al.[69] used potassium permanganate treatment on banana fiber, and flexural strength and modulus of its polypropylene-based composites were found to enhance.
Datta and Kopczynska[70] prepared a solution of 0.5 wt.% $KMnO_4$ in acetone to modify the kenaf fiber for preparing kenaf/polyurethane composites. Joseph et al.[47] used chemical treatments like sodium hydroxide, permanganate, and isocyanate to boost the bonding between low-density polyethylene resin and sisal fiber. Melo et al.[71] performed treatments using alkali, peroxide, potassium permanganate, and acetylation to enhance interfacial bonding to prepare PHB-based biodegradable composites with randomly oriented treated carnauba fibers and noted an enhancement of composites in its mechanical properties.

Scheme 5

Acetyl Treatment

Acetyl treatment is used to modify the structure of natural fiber and also known esterification method. In acetyl treatment, a reaction occurs between the acetyl group (CH_3CO-) the hydroxyl groups (-OH) of the fiber to exclude the moisture; therefore, a reduction in the hydrophilic nature of the fiber takes place. Additionally, after treatment, better mechanical interlocking of fibers with the matrix is observed due to the rough surface generation. Acetylation is carried out on pre-alkali-treated fibers. Acetylation reaction with and without acid catalyst on fiber is shown in Scheme 5. Acetylation treatment of sisal fibre was performed to boost the fibre–matrix adhesion. The methodology incorporated an alkaline treatment at first, trailed by acetylation. Mishra et al.[72] explored the acetylation of sisal fibres by immersing in 5 and 10% NaOH solution for 1 h at 30°C. The alkaline-treated fibre was soaked in glacial acetic acid for 1 hour at 30°C and decanted and soaked in acetic anhydride containing one drop of concentrated H_2SO_4 for 5 minutes.



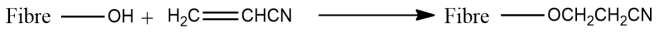
Scheme 5

It was also revealed that acetylated natural fibre-reinforced polyester composites displayed superior bio-resistance and less tensile strength than composites with silane-treated fibre in biological tests.[73]

Acrylation and acrylonitrile grafting

To boost the mechanical interlocking of fiber with matrix acrylic acid (CH_2=CHCOOH) is used. The treatment procedure involves applying concentrations of acrylic acid on alkali pre-treated fibers.[26] The reactions between fiber OH groups and acrylic acid are given in Scheme 6 and 7.

Scheme 6

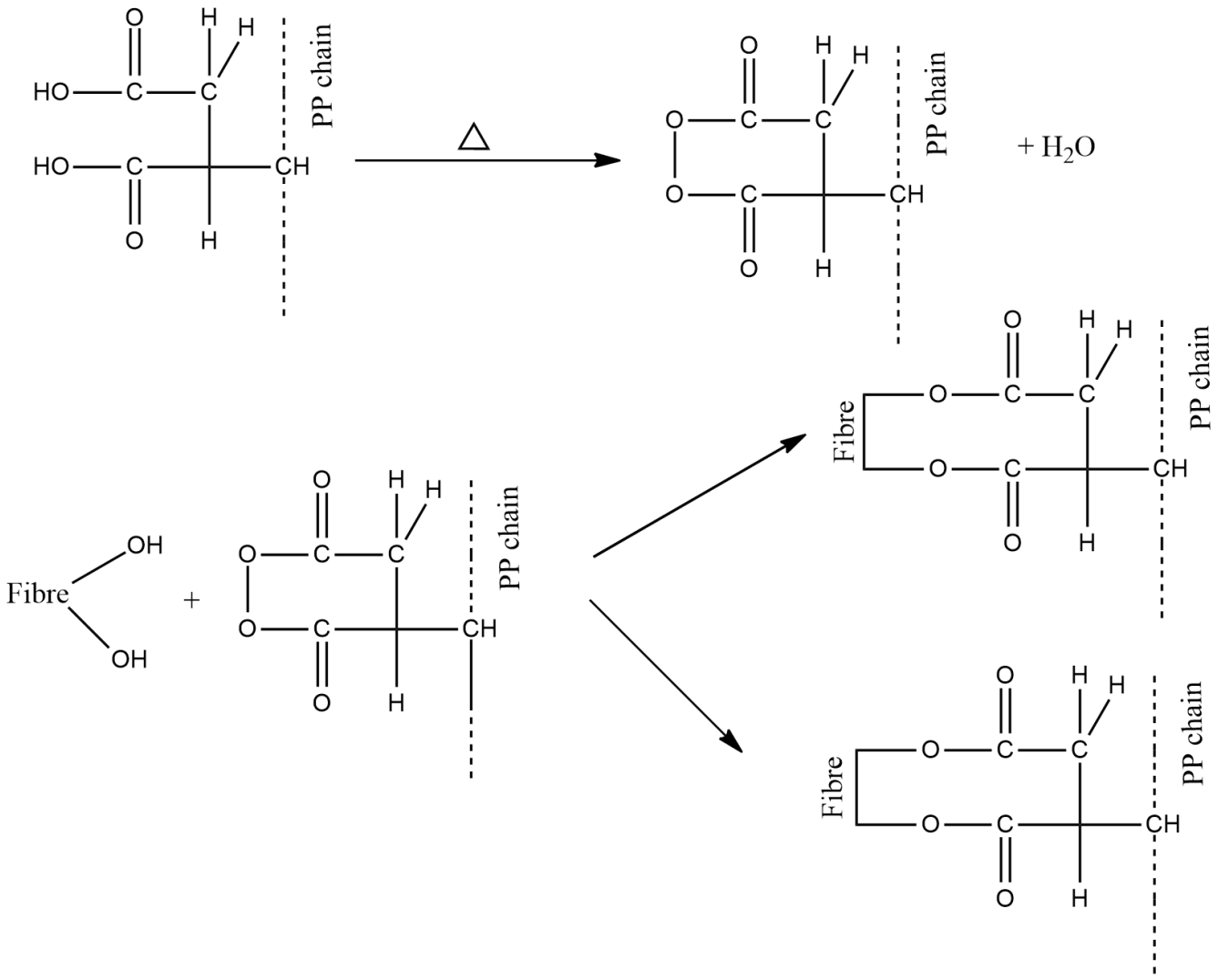


Scheme 7

Sreekala et al.[63] modified oil palm fibres with 10% NaOH for about 30 minutes and then treated with an acrylic acid solution at 50°C for 1 hour at various concentrations. The fibres were washed with an aqueous alcohol solution and dried. The tensile strength of oil palm fibre–PE composites did not increase. Treatments like silane and acrylation led to the strong covalent bond formation, and thereby, the tensile strength and Young's modulus of treated fibers were improved possibly.

2.8 Maleated coupling agents

Effective interaction between the matrix and the fiber is found using maleated coupling agents for surface modification.[74,75] Maleic anhydride reacts with the hydroxyl groups in the amorphous region of natural fiber. Long-chain polymer coating was produced on the fiber surface by using maleated coupling agents to reduce the hydrophilicity.[76] The reaction mechanism of maleic anhydride, polypropylene (MAPP), and cellulose fiber are presented in Scheme 8.



Scheme 8

Concluding Remarks

Many research works on the mechanical properties of biocomposites have been made. It was evidenced that the mechanical properties of these composites depend on the various parameters such as properties of matrix and reinforcement, fiber's shape and size, fibers content, compatibility and wettability, interfacial bonding, and manufacturing methods. In addition, a great deal of work has been done to analyze the influence of chemical treatments on the properties of biocomposites.

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