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Cellulosic Fibril–Rubber Nanocomposites

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8.1 Introduction

The interest in developing composite materials containing nanoreinforcement has grown tremendously in recent years. The main advantages are the attractive properties due to the nanometric size of reinforcement. There are two reasons for changes in material properties when the size of the reinforcing phase is reduced down to the nanometer range:

1. The large surface area associated with nanoparticles results in many interfaces between the constituent intermixed phases that play an important role on the macroscopic properties. In addition the mean distance between particles is much lower as their size is reduced, favoring particle/particle interactions.
2. The occurrence of possible quantum effects, viz. changes in magnetic, optical or electrical properties.

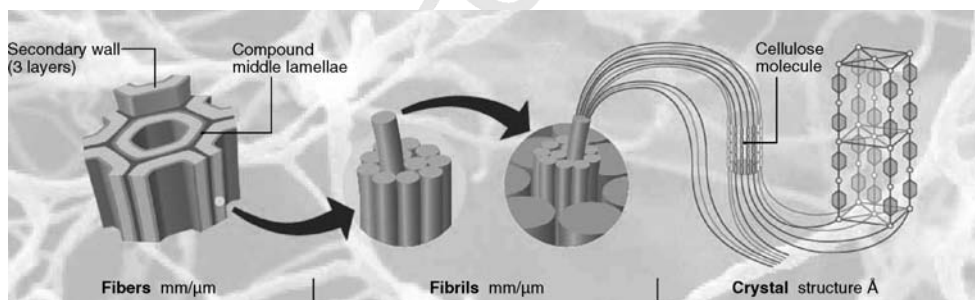
When the reinforcing particles are cellulosic materials, there are additional features like biodegradability and renewability, along with the inherent stiffness and high degree of crystallinity. It is also relatively inexpensive and has a much lower density than most fillers that are in use today. The main challenge has been in getting a good dispersion without any agglomerates in a continuous matrix. Another problem lies in the tedious processing steps by means of purification, bleaching, fibrillation and hydrolysis. There are different techniques for

1 the isolation of cellulose whiskers. Acid hydrolysis of cellulose removes amorphous regions
2 and has been adopted by several researchers. Researchers at CERMAV-CNRS have separated
3 cellulose from various sources like wheat straws and tunicin and have used as reinforcements
4 in polymer matrices [1, 2]. Winter of Cellulose Research Institute at ESF found that the
5 addition of an ounce (28.35 g) of cellulose nanocrystal to a pound (0.45 kg) of plastic resulted in
6 a 3000-fold increase in strength [3].

7 This chapter provides an outlook into nanoreinforcements like cellulosic nanofibers and its
8 reinforcement effects in rubber composites.

10 8.2 Cellulose

12 Cellulose is the main constituent of plant structures, bacteria (e.g., *Acetobacter*) and tunicates.
13 The annual production of cellulose is about 1.56×10^{12} t [4]. The chain conformation and
14 microfibrillar morphology contribute to a significant load-carrying capability. The axial
15 Young's modulus of cellulose has been measured to be 137 GPa [5], which is similar to
16 aramid fibers. The cellulosic units have a complex, layered structure consisting of a thin
17 primary wall that is the first layer deposited during cell growth encircling a secondary wall. The
18 secondary wall is made up of three layers and the thick middle layer determines the mechanical
19 properties of the fiber. The middle layer consists of a series of helically wound cellular
20 microfibrils formed from long-chain cellulose molecules: the angle between the fiber axis and
21 the microfibrils is called the microfibrillar angle. Such microfibrils have typically a diameter
22 of about 10–30 nm, are made up of 30–100 cellulose molecules in extended chain conformation
23 and provide mechanical strength to the fiber. Figure 8.1 represents the structure of the cellulosic
24 cell wall [6].



37 **Figure 8.1** Structure of a cell wall [6]

39 Chemically cellulose is a natural polymer consisting of D-anhydroglucose ($C_6H_{11}O_5$)
40 repeating units joined by β -1,4-glycosidic linkages at C_1 and C_4 position [7]. The degree
41 of polymerization (DP) is around 10 000. Each repeating unit contains three hydroxyl groups at
42 C-2, C-3 and C-6 linkages. Cellulose does not dissolve in common solvents or water at its
43 ordinary state because it crystallizes by intra- and intermolecular hydrogen bond linkages. The
44 presence of these hydrogen bond linkages is responsible for the chain stiffness and stability of
45 the conformation of cellulose. These hydroxyl groups and their ability to hydrogen bond play a
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major role in directing the crystalline packing and also govern the physical properties of cellulose. Solid cellulose forms a microcrystalline structure with regions of high order (i.e., crystalline regions) and regions of low order (i.e., amorphous regions). Cellulose is resistant to strong alkali (17.5 wt%) but is easily hydrolyzed by acid to water-soluble sugars. Cellulose is relatively resistant to oxidizing agents. Although starch has the same basic structure as cellulose – it is also a polysaccharide – the glucose subunits are bonded in such a way that allows the starch molecule to twist. In other words, the starch molecule is flexible, while the cellulose molecule is rigid.

Cellulose exists in several crystal modifications. Naturally occurring cellulose is known as cellulose I which exists in parallel strands without intersheet hydrogen bonding. Cellulose II is thermodynamically more stable and exists in antiparallel strains with intersheet hydrogen bonding. The difference in properties of cellulose I and II arises due to changes in crystal structure [8]. Table 8.1 shows some of the physical properties of cellulose I and II [9].

Cellulose III is amorphous and obtained by treatment of cellulose I or II with amines. Cellulose IV is obtained after treatment of cellulose III with glycerol at very high temperatures. Deguchi *et al.* [10] reported that cellulose undergoes a crystalline to amorphous transition in water at 320 °C and 25 MPa. The transformation is associated with a large change in mechanical and chemical properties, just like the gelatinization of starch. The authors predict that this newly found property of the most abundant and renewable biomass is of significance to its utilization, for example, biomass conversion.

Table 8.1 Some important physical properties of cellulose I and cellulose II

Property	Cellulose I	Cellulose II
DP	103–104	250–240
X-ray crystallinity (%)	50–75	25–40
Density (g cm ⁻³)	1.53–1.89	1.49–1.55
Breaking strength (dry) cNtex ⁻¹	26–50	14–61
Elastic modulus (dry) cNtex ⁻¹	2–11	8–40
Water vapor regained at 65% relative humidity (%)	7–8	12–14

8.3 Cellulosic Nanoreinforcements

8.3.1 Cellulosic Microfibrils

Cellulose microfibrils can be separated by chemical and mechanical treatments. The diameter of cellulose microfibril is about 5–10 nm and the length varies from 100 nm to several microns, depending on the source. Each microfibril consists of monocrystalline cellulose domains linked by amorphous domains. On acid hydrolysis the microfibrils undergo transverse cleavage along the amorphous regions into microcrystalline cellulose or whiskers. The perfect crystalline arrangement of whiskers results in a high modulus and makes them act as efficient reinforcing materials [11–13].

Figure 8.2 represents the microfibrillar arrangement in cellulose [14]. Despite a high Young's modulus, there are problems associated in realizing the full potential of the microfibrils, as the size of agriculturally based fibers varies depending on the isolation procedure and cellulose source. Second, the disintegration of cellulose from a plant cell wall at a reasonable cost and

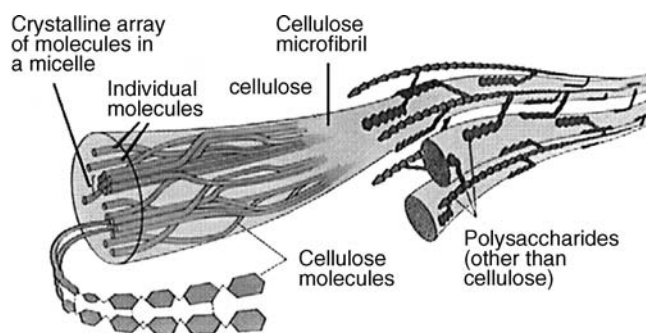


Figure 8.2 Microfibrillar arrangement in cellulose [14]

without severe degradation is a problem. Third, the dispersion of microfibrils in a polymer matrix is difficult as the high density of hydroxyl groups at the microfibril surface induces strong interaction between the microfibrils and they tend to agglomerate [15].

Cellulose microfibrillar surfaces also provide potential for surface modification using well established carbohydrate chemistry [16]. Stenstad *et al.* [17] reported on the chemical modifications of microfibrillated cellulose (MFC) and observed that the surfaces of MFC could be activated and functionalized in both aqueous and organic solvents. The surface modifications used were the grafting of hexamethylene diisocyanate, succinic acid and maleic acid. Alkali treatment was used by Nakagaito and Yano [18] to enhance the toughness of microfibrillated cellulose-reinforced phenolic composites. The improvement was attributed to the transformations in the amorphous regions along the cellulose microfibrils.

In an interesting study, cellulose nanofibril whiskers were synthesized from banana fibers by the process of steam explosion in alkaline medium followed by acidic treatment. This method was found to be very effective in the depolymerization and defibrillation of the fiber to produce banana nanowhiskers. In this study, the authors [19] adopted steam treatment with subsequent explosive defibrillation. This was followed by characterization with X-ray diffraction (XRD) studies and transmission electron microscopy (TEM). XRD studies revealed a reduction in fiber size and an increase in fiber crystallinity. Figure 8.3 shows the TEM of steam exploded banana fibers in acidic medium revealing a needle like structure. The average diameter of the nanofibrils was found to be 4–5 nm and the average length of nanofibrils to be 200–250 nm.

Recently researchers have developed cellulose nanopaper from wood fibrils of high toughness [20]. The high toughness of highly porous nanopaper is related to the nanofibrillar network structure and high mechanical nanofibril performance. The nanopaper exhibits a large strain-to-failure, which indicating mechanisms, such as interfibril slippage, which contribute to inelastic deformation in addition to deformation of the nanofibrils themselves. Figure 8.4 shows a fibrous nanofibril network film which is fine and web-like, with a highly fibrous network structure. The nanofibril length is several microns and nanofibril ends are not apparent. Furthermore, individual nanofibrils are swirled and physically entangled with respect to each other. The nanopaper sample shows very high toughness, the work to fracture being 15 MJ m^{-3} , in uniaxial tension and this was associated with a strain-to-failure as high as 10%. The Young's modulus (13.2 GPa) and tensile strength (214 MPa) were remarkably high although there was a high porosity of 28%.

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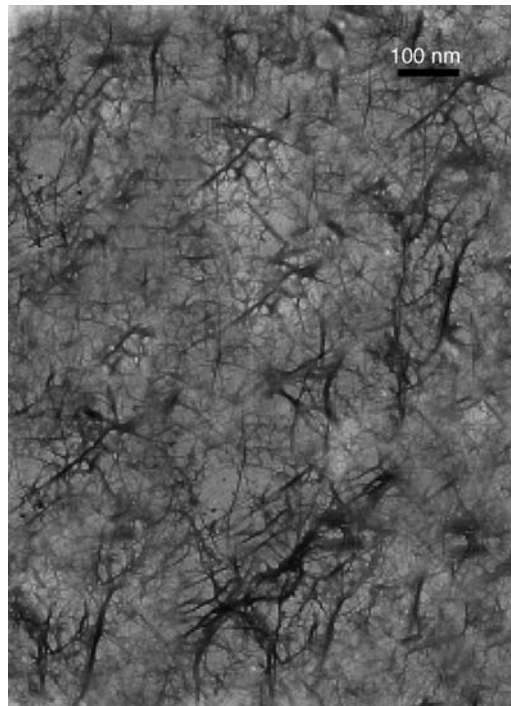


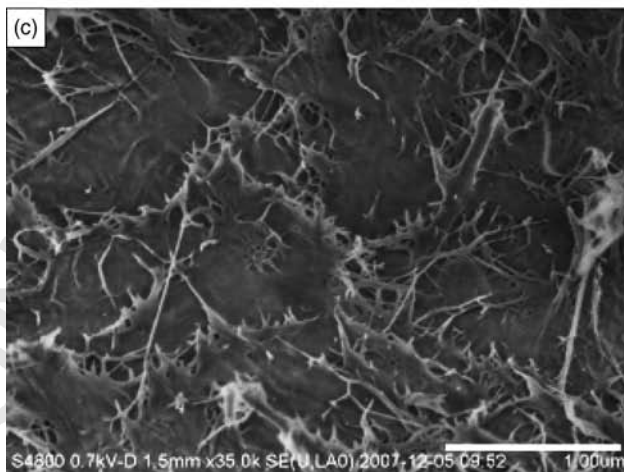
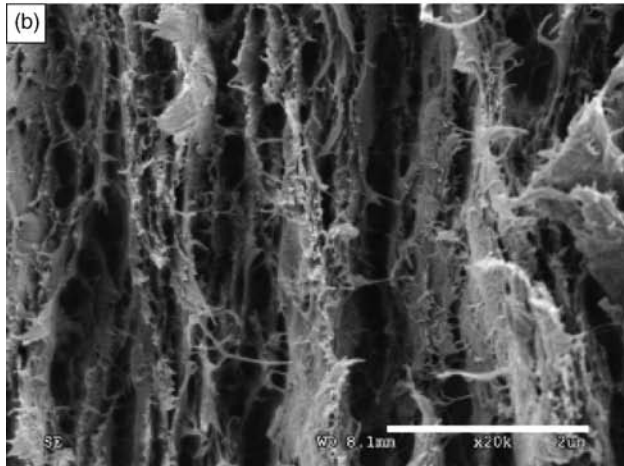
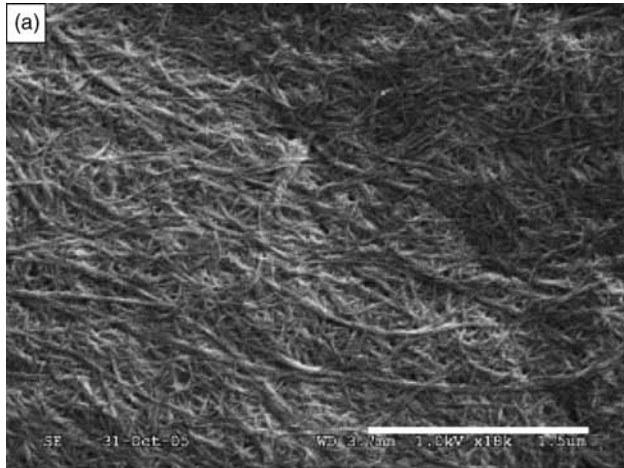
Figure 8.3 Transmission electron micrograph of steam-exploded banana fibers in acidic medium [19] (Reproduced with permission from B.M. Cherian, L.A. Pothan, T. Nguyen-Chung, G. Mennig, M. Kottaisamy and S. Thomas, “A Novel Method for the Synthesis of Cellulose Nanofibril Whiskers from Banana Fibers and Characterization,” *Journal of Agricultural and Food Chemistry*, **56**, no. 14, 5617–5627, 2008. © 2008 American Chemical Society.)

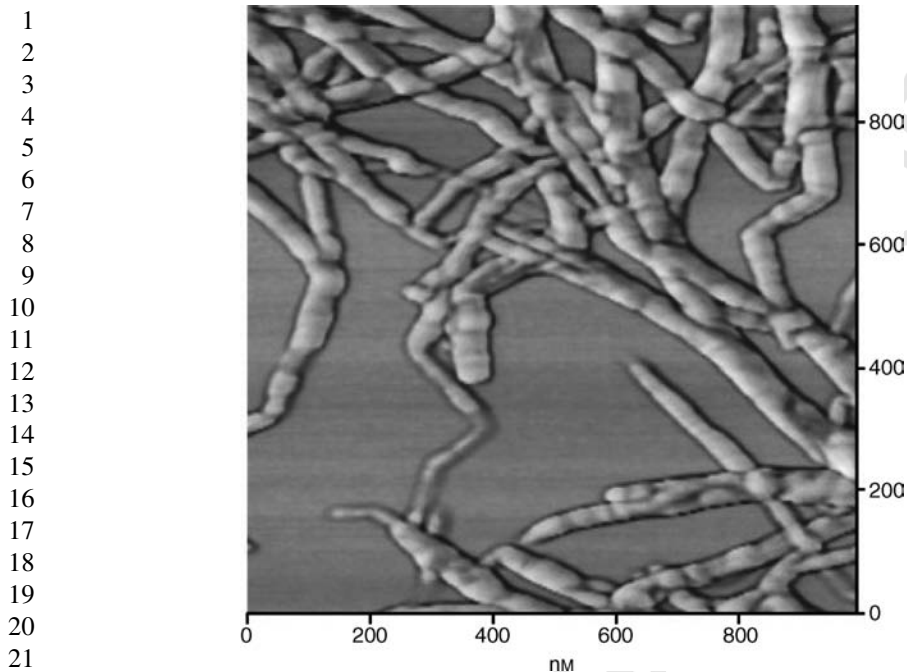
Zuluaga *et al.* [21] isolated and characterized cellulose microfibrils from banana rachis using a combination of chemical and mechanical treatments. The morphology and structure of the samples were characterized using transmission electron microscopy, atomic force microscopy and X-ray diffraction. Suspensions of bundled or individualized 5 nm microfibrils were obtained after homogenization whereas an organosolv treatment resulted in shorter aggregates of parallel cellulose microcrystallites. It was also found that microfibrils and microcrystals prepared by both methods can be used as reinforcing filler in nanocomposite materials.

Bhattacharya *et al.* [22] successfully isolated and characterized cellulose microfibrils from bagasse fibers. Bagasse fibers were subjected to conventional pulping to eliminate lignin and hemicellulose. The fibers obtained were separated into constituent microfibrils by a two-stage homogenization process and finally acid hydrolyzed. Atomic force microscopic (AFM) studies revealed that the transverse size of the particles varied between 200 nm to a few microns. Figure 8.5 shows the AFM of the microfibrillar bundles composed of 30 nm nanofibers.

Abraham *et al.* performed a series of experiments in extracting cellulosic nanofibers from various natural fibers. The natural fibers chosen were coir, banana, sisal and pineapple. The fibers were characterized by different techniques like scanning probe microscopy and

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23 **Figure 8.5** Microfibrillar bundles are also observed to be composed of nanofibers (~ 30 nm) [22]
24 (Reprinted from *Carbohydrate Polymers*, **73**, D. Bhattacharya, L.T. Germinario and W.T. Winter,
25 “Isolation, preparation and characterization of cellulose microfibrils obtained from bagasse,”
26 371–377, © 2008, with permission from Elsevier.)

28 XRD [23]. The scanning probe micrographs (SPM) of modified fibers are given in Figure 8.6.
29 The results reveal that fiber diameter can be reduced to the nanometer range using steam
30 explosion coupled with acid hydrolysis. Table 8.2 shows the values of the crystallinity index
31 obtained in the case of variously treated fibers. XRD analysis of the alkali-treated fibers
32 revealed an increase in the crystallinity index of the banana and coir. An improvement in the
33 order of the crystallites was observed as the cell wall thickened upon alkali treatment. The
34 crystallinity index initially increased but then declined at high alkali concentrations when
35 damage to the cell wall occurred.

36 Recently researchers have successfully isolated nanofibers from soybean and analyzed its
37 reinforcing capacity in polymers [24].

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40 **Figure 8.4** FE-SEM micrographs of: (a) a cellulose nanofibril film surface showing a fibrous network
41 (scale bar is $1.5\ \mu\text{m}$); (b) the cross-section of a fracture surface of a film showing a layered structure (scale
42 bar is $2.0\ \mu\text{m}$); and (c) a fracture surface viewed perpendicular to the film surface (scale bar is $1.0\ \mu\text{m}$).
43 These films were dried from water suspension. The film in (a) was prepared from DP-1100 and the other
44 two were prepared from DP-800 [20] (Reproduced with permission from M. Henrickson, L.A. Berglund,
45 P. Isaksson, T. Lindstrom and T. Nishino, “Cellulose Nanopaper Structures of High Toughness,”
46 *Biomacromolecules*, **9**, 1579–1585, 2008. © 2008 American Chemical Society.)

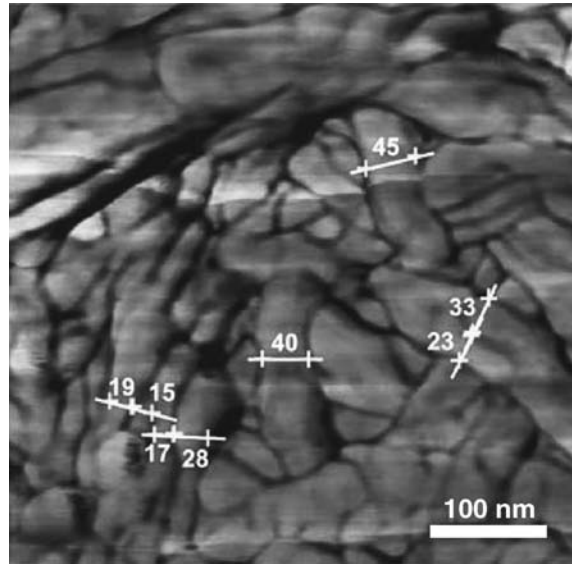


Figure 8.6 Scanning probe micrographs of modified banana fibers [23]

Table 8.2 Crystallinity index values of treated fibers

Fiber stage	$I(0,0,2)$	I_c (%)
Raw banana fiber	$I(0,0,2) = I_{amo} = 10.5$	—
Steam-exploded banana fiber	16.4	35.97
Steam-exploded bleached banana fiber	22.9	54.18
5% oxalic acid-treated bleached banana fiber	31.1	66.23
Raw coir fiber	$I(0,0,2) = I_{amo} = 4.96$	—
Steam-exploded coir fiber	5.19	4.43
5% oxalic acid treated bleached coir fiber	17.7	71.97

8.4 Studies on Cellulosic/Latex Nanocomposites

A survey of the literature has shown that studies on cellulosic nanofiber-reinforced latex composites have been mostly unexplored. With the exception of studies conducted by research groups in France and recently in India, only a limited amount of work is reported in literature.

Orts *et al.* [16] investigated the reinforcements effects of cotton microfibrils at a concentration of 2.5% in latex emulsion. It was observed that the maximum load increased several-fold and the percent elongation at maximum stress increased twofold. Similar results were also observed for latex composites reinforced with straw derived microfibrils [25, 26]. Figure 8.7(a) and (b) presents the variation of load and elongation at break on the addition of 2.5% cotton microfibril to latex emulsion.

Favier *et al.* [27] demonstrated the benefits of reinforcing a polymer with cellulose whiskers. The authors used 6% cellulose whiskers derived from tunicate cellulose in a latex polymerized

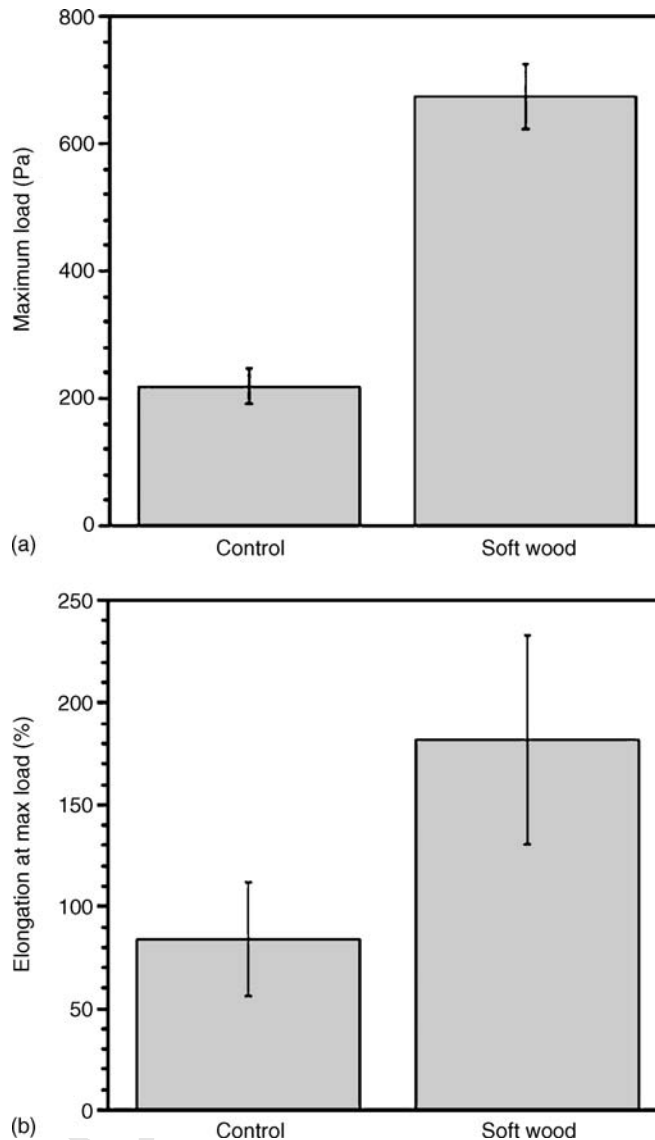


Figure 8.7 Effect of the addition of 2.5% cotton microfibril to a filmcast from latex emulsion. Maximum load (a) and percent elongation at maximum load (b) are shown for untreated and microfibril-reinforced film [16] (With kind permission from Springer Science + Business Media: *Journal of Polymers and the Environment*, “Application of Cellulose Microfibrils in Polymer Nanocomposites,” 13, no. 4, © 2005, 301–306, W.J. Orts, J. Shey, S.H. Imam, G.M. Glenn, M.E. Guttman and J.W. Revol.)

from styrene and butyl acrylate and observed that the composite films exhibited a twofold increase in the shear modulus over control films containing no whiskers. The whiskers were found to form a rigid network linked by hydrogen bonds. The simulation and modeling of the structures were also reported [28].

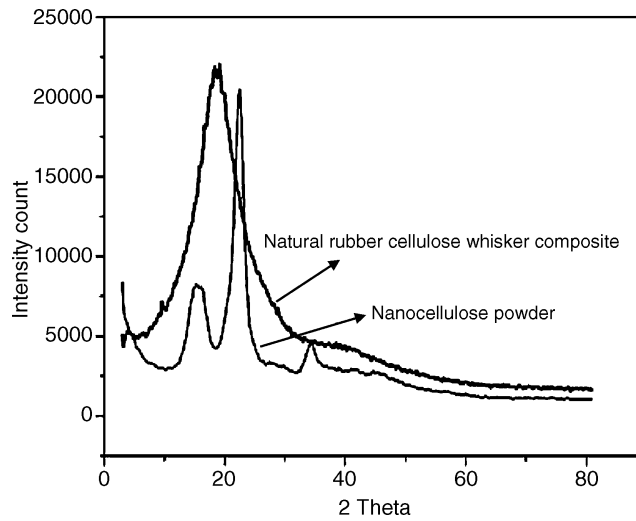


Figure 8.8 XRD curves of the natural rubber/nanocellulose composite [23]

Hajji *et al.* [29] reported on the tensile behavior of nanocomposites from latex and cellulose whiskers observed significant improvement. The thermomechanical properties of these nanocomposites were investigated, and the influence of processing conditions and the effect of whisker content were considered. Processing conditions were found to have a large influence on the mechanical behavior and can be classified in ascending order of their reinforcement efficiency: It can be attributed to a decrease of the apparent whisker aspect ratio, due to gradual breakage and/or orientation of the whiskers when hot pressing or extrusion is used. Above the glass transition temperature a reinforcing effect was observed, which was related to the presence of a rigid cellulose network, linked by hydrogen bonds.

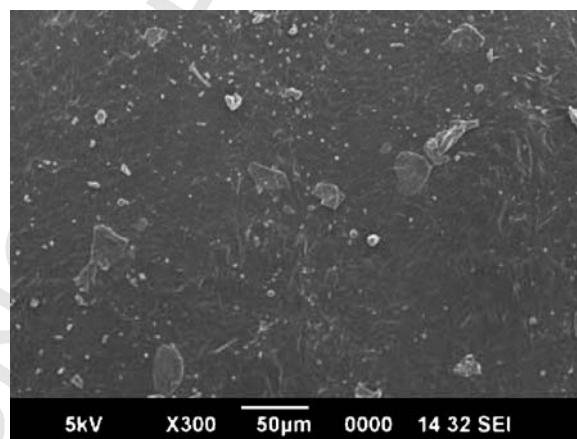


Figure 8.9 SEM of nanocellulose whisker in the natural rubber latex [23]

1 Recently banana nanofibers obtained by the process of steam explosion were incorporated in
2 matrices like PLA and natural rubber latex to form composites [30]. The main feature observed
3 in the natural rubber composite was a high increase in the mechanical properties after
4 incorporating the nanocellulose. Due to the uniform dispersion of the nanocellulose in rubber
5 latex the prepared composites showed improved stiffness with out any loss of its elastomeric
6 nature. The stress was higher for all systems containing nanofiber composites than the pure
7 natural rubber sample. This is an indication of the effectiveness of reinforcement. XRD
8 analysis of the natural rubber/nanocellulose composite showed the dispersion of nanolayers of
9 cellulose in the polymer matrix (Figure 8.8). The dispersion of the nanocellulose was further
10 confirmed by SEM (Figure 8.9).

11 12 13 **8.5 Conclusions**

14 This chapter presents an overview of studies on cellulosic nanofiber-reinforced rubber
15 composites. The properties of cellulose and cellulosic microfibrils have been highlighted.
16 Though sufficient data is present on cellulosic fiber-reinforced polymer nanocomposites,
17 studies pertaining to rubber composites are few and need to be addressed in detail. The
18 problems lie in the tedious process of extracting nanofibers from cellulosic sources and their
19 uniform dispersion in a continuous matrix. By addressing certain scientific and technical
20 challenges the possibility of advanced structural materials based on cellulosic nanofibers is
21 likely to be a reality in the near future.

22 23 24 **References**

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Abstract

Cellulose – the most abundant polymer on earth – has emerged as an ideal candidate for providing nanoparticles as reinforcing agents. There is a growing interest in cellulose nanocomposites within the research community and especially if the composites are based on renewable resources. This chapter deals with various aspects of cellulosic nanofiber-reinforced rubber nanocomposites. A brief description of cellulosic microfibrils and its properties is mentioned and some of recent studies dealing with cellulosic fibril-reinforced rubber composites are highlighted.

Keywords: cellulose, microfibrils, rubber, nanocomposite, latex, mechanical properties, natural fibers, surface modification

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